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# CHEMICAL AGE

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## UK CHEMICAL EXPORTS

THE decline in UK chemical exports is not so serious as the Trade and Navigation Accounts for the first half of the year would suggest. Apart from the dock strike which affected shipments in both May and June, last year's total chemical exports of £267 million were the highest ever. It was obvious at the beginning of the year that the US recession, coupled with a general stiffening in world competition and surplus capacity for some basic chemicals, would inevitably mean a falling off in the overseas trade of British chemical manufacturers.

It is remarkable that this has not been higher. For the first five months of the year exports were 3.5 per cent lower than a year ago and for the first six months were lower by 5.4 per cent. That export performances in both months were affected by the dock strike is clear when figures are compared with the monthly average decline for 1957. Exports in May were worth £22,846,439, a drop of £2,319,393 compared with May 1957—this decline is much greater than the monthly average decline which for the first five months was £765,033. Similarly, exports in June, worth £17,800,553, showed a fall of £3,494,732 over June, 1957, a decline that considerably exceeded the monthly average fall for the first half of the year, which was £1,214,526. Doubtless, figures for subsequent months will show a recovery.

For the five months to May, UK chemical shipments totalled £110,942,799, and were £3,825,167 below the first five months of 1957. Exports in the first half of this year, at £128,785,592, were £7,287,156 below the same period of 1957.

Dealing with exports in the first half of the year, it should be noted that the decline did not apply to all chemicals. Many did not conform to the general pattern and showed increases compared with the same period a year ago. In inorganic chemicals, slight increases were recorded for acids, ammonia, calcium compounds, cobalt compounds and sodium compounds; while there was a substantial increase in shipments of carbon blacks, which rose from £1,388,008 to £1,760,350 in the six months period.

Exports of inorganic chemicals which fell in the same period included copper sulphate, at £1,185,251, down by £760,000; sodium hydroxide (caustic soda), at £1,889,545, down by £1,296,000; soda ash, at £1,134,372, down by £516,000; sodium arsenical compounds, halved at £85,432.

In the field of organic chemicals, shipments of glycerine, at £207,581, were more than halved by a fall of £295,772; salicylates, at £95,109, almost halved, and organic dyestuffs and intermediates, at £425,110, down by £336,000. In organic chemicals, rises were recorded by acids, anhydrides, their salts and esters, acetone, phenol, sulphonamides (not prepared) and compressed, liquefied or solid gases, which at £1,466,382 showed a substantial rise of £405,000.

Total exports of mineral tar and crude chemicals from coal, petroleum and natural gas, for the first half of the year were £1,469,348, representing a fall of £449,000. In this field exports of coal tar, at £374,065, were down by £114,000; and cresylic acid, at £303,260, down by £331,000; while creosote oil shipments, totalling £650,603, were up by £111,000.

Shipments of synthetic organic dyestuffs and compounds, but not including organic intermediates, were worth £4,492,132, a decline of £956,000.

In the drugs and pharmaceuticals section, the first half-year shipments of antibiotics were down slightly from £4,257,540 to £4,186,980. Figure for the whole section totalled £18,495,553, a drop of £1,331,889.

## DEVELOPMENTS IN PLASTICS

**R**ECENTLY further developments have been announced in plastics. Reichhold Chemicals, for example, have reported within the last three weeks that they can now make an unmodified phenolic resin for low and high pressure laminates that will withstand temperatures up to 4,500° F. for short periods, and up to 500° F. for over 100 hours. One application for this resin, the company believes, will be in military missiles. No details have been released regarding the method of preparation other than that the process technique and catalyst system used are the main factors. A price has, however, been worked out—namely, 67 cents per pound in quantity.

From the Tank Lining Corporation comes news of a new technique of applying polythene to steel tanks, pipe and valves, without any alteration of the plastics materials properties. (*Chem. and Engng. News*, 1958 **36**, No. 28, 44). To apply the polythene, the steel surface is prepared, the polythene is sprayed on and is heat treated to cure it. As a patent is to be applied for, process details are being kept dark, but available information indicates that the success of the process is tied up with the polythene used (described as a specially prepared low pressure process material). Heat treatment is of importance, careful control of temperature being necessary during curing by heating in an oven or, for large pieces of equipment, by hot air.

Tests carried out by the company are stated to have shown no chemical or physical changes in the properties of polythene. That is, tensile strength, elongation, flexural strength, thermal expansion, thermal conductivity, flammability, non-absorption of water, moisture vapour permeability and dielectric strength. Electrical insulation properties of the lining are reported as good, and abrasive resistance moderate. Heavy films can be used and the lining produced is flexible and resilient.

Polythene has been developed as a purging resin by US Industrial Chemicals, its purpose in this instance being to reduce waste caused by shutting down polythene extrusion and injection moulding machines. Resin deterioration is usually observed, sometimes for hours, after starting-up a machine that has previously operated on conventional polythene before shut-down. The reason for this is considered to be due to the plastics material staying too long at too high a temperature in some parts of the machine.

The special USI resin is Petrothene 205-1. A few minutes before the shut-down is due, the purge resin is fed to the machine. The normal resin is purged and the machine can be shut down with no waste and no after affect.

An electrodeposition process has been developed by the Redel Company, US, by means of which Teflon (polytetrafluoroethylene) is deposited on metallic and non-metallic parts and is then fused to form a coating. It is claimed that this process gives better adhesion, less porosity, and better control of coating thickness than conventional dispersion techniques. Applications for the process are valve stems and bodies, and electronic components.

Now marketed by Semet-Solvay Petrochemical division is an emulsifiable polythene for use as a textile finishing agent. Given the trade name A-C Polyethylene 629, it is claimed that it overcomes loss of hand and tear strength when used with thermosetting resins common for finishing wash-and-wear garments.

## NEW STEROIDS

**D**ETAILS have been released by Merck Inc., US, of a new steroid compound, hexadecadrol (16 $\alpha$ -methyl 9- $\alpha$ -fluoroprednisolone) marketed as Decadron, which is

claimed to be 30 times more potent, from preliminary chemical trials, than hydrocortisone, itself seven times more potent than prednisone and prednisolone.

The high potency is due to the methyl group at the 16- $\alpha$  position in the D ring of the steroid molecule. Starting material is an intermediate from Merck's hydrocortisone process. A methyl group is added at C-16 in an unsaturated 20-keto pregnene by means of a Grignard reaction with methyl magnesium iodine (*JACS* 20 June 1958). After other steps, epoxidation, dehydrogenation and introduction of fluorine, Decadron is evolved.

Prednisone, prednisolone, hydrocortisone and Squibb's 9- $\alpha$ -fluorohydrocortisone have all been modified with a methyl group at C-16. All these compounds have proved to be highly potent in clinical use. Merck suggest that the 16-methyl group on the D ring acts as a protection for the active centres of the steroid molecule, thereby preventing metabolic breakdown and giving enhanced potency.

Clinically all these methyl-modified steroids have proved to be more potent than hydrocortisone, but from trials already conducted hexadecadrol appears to be the most potent yet. At the same time, the new compound does not seem to give rise to such untoward effects as sodium and water retention or potassium loss and to produce no oedema. Merck hope to market the drug next year.

Another potent new cortisone-like hormone has also been announced by Upjohn Co., Kalamazoo, Michigan. This new compound is 6 $\alpha$ -9 $\alpha$ -difluoroprednisolone and is reported as being 400 times as active as hydrocortisone in preliminary tests with rats.

An important chemical step in the preparation of the new material is the synthesis of 6- $\alpha$ -fluoroprednisolone just announced by both Upjohn and Syntex SA.

The first synthesis of the female sex hormone equilin is also reported from the US. This has been accomplished by Dr. C. Djerassi (Syntex). Interest here centres on the fact that this substance is a naturally occurring, physiologically active steroid hormone which until now has resisted all attempts at partial or total synthesis. The very limited supplies of equilin have all been obtained from pregnant mares' urine. The synthesis involves 11 steps starting from testosterone.

## PICKLE LIQUOR DISPOSAL

**T**ECHNICAL success of the Ruthner process for converting waste pickle liquor has been reported in a recent report by the sponsors' committee to the American Iron and Steel Institute. The Ruthner process development programme was set up by Blaw-Knox and a consortium of seven steel producers at a cost of some \$500,000. A 1,440-gallon-a-day pilot plant in Ohio has shown that the process is technically a success, particularly in overcoming certain severe corrosion problems (*Chemical Engineering*, 1958, **65**, No. 13 p. 54).

In the Ruthner process, waste pickle liquor is converted into reusable sulphuric acid via formation of a ferrous chloride intermediate, which is then roasted to produce commercially valuable iron oxide and recycle HCl.

The steel producers now have to decide, it is reported, if, when and how to utilise the process. Opinions among the producers appear to be divided as to the technicalities of the process and to the reliability of cost estimates. At the present time, despite credits for recovered acid and iron oxide at current (US) prices, the Ruthner plant would still be a losing proposition, it is stated. However, it seems that if consideration is given to the best disposal process for prevention of pollution, Ruthner process has decided advantages over lime neutralisation and other techniques.

It is suggested that the US steel producers may erect a commercial scale Ruthner plant to process the waste pickle liquor from a group of steel mills.

# INDUSTRY AND ATOMIC ENERGY

## AEA Report Gives News of Development in Fuels, Isotopes and Instruments

**M**ORE details than have previously been found in UK Atomic Energy Authority reports are contained in the fourth annual report, 1957-8 (HM Stationery Office, price 4s 6d). The 68 page booklet includes chapters on raw materials, processing plants, work on controlled thermonuclear reactions, and on many aspects of research carried out by the AERE, Research and Development Branch, Industrial Group, and Weapons Group.

At a press conference Sir John Cockcroft last week stated that the Authority had 'plenty of ideas and plans in the shape of laboratory research'. Now that the Winfrith establishment was being developed it was not thought likely that any more sites would be needed in the immediate future—the area in Scotland which had been looked at would not now be required.

Sir Edwin Plowden, chairman of UK AEA, agreed that, if Britain built gas cooled reactors using enriched fuel, it might prove economical to import some of the fuel from the US, whose facilities were better for production of this material. Construction costs of such plants would, however, be lower. The present 'glut' of uranium would probably not reduce prices to the Authority until at least the middle 1960's, since present contracts generally carried on till then. Between April 1962 and March 1963 the Authority will purchase about \$105 million worth of uranium concentrates from the Canadian government's selling agency, but S. Africa continued to supply much the greater part of the imports during 1957-8.

### Pilot Plant for UF<sub>4</sub> Nearly Complete

**Process Developments.** Much work has been done on process developments, particularly at Springfields, Windscale and Capenhurst. During the year further refinements to the solvent extraction purification process and the magnesium reduction process have been developed: the main object was now to establish a fluidised bed process for the conversion of the pure uranyl nitrate solution from the former to uranium tetrafluoride suitable for the latter. In a small scale plant, several tons of UF<sub>4</sub> have been made and reduced to metal successfully. A full-scale pilot plant, of which the construction is nearly complete, has been designed for the process. The first stage of the reconversion of uranium hexafluoride to metal (ie. its conversion to uranium tetrafluoride) has already been carried out at Capenhurst for over a year.

At Windscale equipment for extracting protactinium from residues arising from the processing of Congo uranium ores is being assembled. At the same station preliminary flowsheets have been drawn up for a plant which will combine the operations of the primary separation, plutonium purification and uranium purification plants, using a single solvent, and also the denitration of uranyl nitrate. It is thought that this system will reduce radioactive effluent disposal problems, and will enable economies to be made in nitric acid utilisation. Other developments at Windscale include: experimental plant work on the extraction of caesium-137 from fission products, based on precipitation followed by ion-exchange; application of theoretical assessments that in the final stages of plutonium production continuous processing would provide economic advantages.

At Capenhurst a plant for the separation of the boron isotopes (the B<sub>10</sub> isotope has powerful neutron absorbing properties) has been operated successfully.

### Applications of $\gamma$ -radiation in Industrial Chemistry

**Radiation and Radioisotopes.** Main work being carried out at the Wantage Radiation Laboratory is concerned with the application of gamma radiation to induce physico-chemical effects of industrial and biochemical importance. The initial work in the field of industrial chemistry has been on polymerisation: a suitable polymer has been grafted experimentally to the material of synthetic fibres to make a permanent conducting film which cannot be removed by ordinary treatments. It is also hoped that, by growing a suitable graft polymer on to motor tyre cords, the adhesion of the cord to a synthetic rubber may be improved.

Other applications of radiation and radioisotopes now being studied include prevention of deterioration in food in store (extermination of grain weevils); control of infestation by the release of radiation-sterilised mature insects (present work on the flour-mill moth); the production of long-lived light sources of low intensity by mixing a suitable phosphor with a radioactive gas such as tritium or krypton-85 (a patent application has been for a film incorporating a suitable phosphor and sealed between suitable transparent sheets of plastics).

In a development under investigation the expensive X-ray machine of the usual equipment for X-ray fluorescent spectrometry is replaced by a cheaper radioisotope. The dose rate is lower

from an isotope than an X-ray tube, but it appears that elements of lower atomic number can be detected with the new technique. In one application chromium plating deposit thickness can be measured down to a few millionths of an inch, irrespective of base coatings of other materials. The most promising radioactive tracer method so far discovered for measuring the efficiency of industrial filters is one using sodium-free glass particles containing 0.01 per cent of gold.

Autoradiography has been used to study surface porosity in magnesium alloys and the distribution in depth of antimony in lead alloys for bearings. The value of isotopically labelled compounds in the technology of such chemical as weedkillers, rubber additives, etc., has induced the Radiochemical Centre to synthesise during 1957-8 some 500 compounds containing 20 different isotopes. This work has been extended to include tritium. It is expected that the laboratories for organic chemistry and for handling alpha-emitting isotopes will be completed at the Centre by 1959. Sales of isotopes in 1957-8 increased by more than 20 per cent to a total value of £650,000.

Certain experience of the Weapons Group has proved of importance to the civil reactor programme. AWRE knowledge of the properties of plutonium and beryllium have helped other AEA sections. Accurate determination of the specific heat of plutonium up to its melting point has been found possible, and a pressureless sintering technique has been developed which will contribute to the Industrial Group's research on beryllium as a canning material for reactor fuel elements. Research at Dounreay has covered studies of liquid sodium and sodium-potassium corrosion: methods of reducing the oxygen content, and so the corrosive effect of sodium, have been installed in the fast reactor circuit.

### New Instruments and Equipment Developed

**Instruments and Equipment.** Some experimental work has resulted in the production of instruments and equipment of interest to the chemical and allied industries. The electronics laboratory at Harwell has developed a new triple crystal scintillation counter of high sensitivity for the detection of radioactive occurrences in the air; also a portable scintillation counter for ground use. Capenhurst workers have devised an analogue computer for the rapid survey of the effects of individual factors on the whole plant operation. Mass spectrometers, normally needing to be operated by qualified staff, have been adapted to routine use by semi-skilled operators. A special instrument is being built for uranium analysis which will measure changes in the isotope concentration of one part in 100,000.

**Reactor Projects.** Reactor plans were to be based on the AEA belief that the type most likely to be successful would be an advanced version of the gas-cooled graphite-moderated reactor at present in use at Calder Hall. AEA spokesmen, however, stated that, in case this system did not come up to expectations, work would be continued on at least the following types: gas-cooled heavy-water moderated; fast-breeder; high-temperature gas-cooled. No further work would be done on sodium-cooled graphite-moderated reactors.

The use of nuclear fuel in the form of uranium rods in magnesium cans imposes definite limits on the temperature at which the gas-cooled graphite-moderated reactor can operate. To achieve higher temperatures a new kind of fuel element (uranium-oxide—a powder—heated and compressed) and new canning materials (perhaps beryllium) will be used in a prototype reactor to be built at Winfrith Heath by the middle of next year. The Winfrith programme also includes plans for a high temperature, graphite-moderated,

zero energy reactor to be commissioned in 1959.

Commercially available graphites are highly porous and tests have been made in an effort to overcome the problem of corrosion and mass transfer caused by the presence of oxidising impurities in the coolant.

**Thermonuclear Apparatus.** It was hoped that Zeta II, design of which has just been started, will achieve the 'break even' point, i.e. that as much energy would be created by fusion as was lost by radiation from hot gas. In the new version shock heating compression would probably be used in combination with the present high current heating techniques employed in Zeta I, and the target was a heat of 100 million degrees.

At the 'Atoms for Peace' conference to be held in September at Geneva, Dr. Peter Thonemann, leader of the Zeta group, will present with other members of the Authority papers giving all details of Zeta available at the time of reading. Sir John Cockcroft stated that about 400 papers on various subjects had been submitted by the Authority for this conference and of these some 70-100 would probably be read.

## Increase in Japanese Chemical Fibres Production

A 'REASONABLE estimate' has been made that Japan is now producing about one-sixth of the world's supply of all chemical fibres and about one-tenth of the synthetics (i.e. nylon, the acrylics, etc., not including rayon). Last year Japan manufactured 1,060 million pounds of man-made fibres, being the second largest producer to the US. An enthusiastic home market has helped Japanese companies to outstrip the UK in bulk chemical fibre production and now large quantities are exported.

Previously, great emphasis in Japan has been given to the cheaper qualities of man-made fibres, and particularly to spun rayon, but polyacrylonitrile fibres are becoming increasingly popular.

Materials somewhat similar to Orlon and Acrilan are being made under the names of Cashmilon, Exlan and Vonnell. Also high-bulk yarns, a relatively new development, are available from three firms, and five other firms will be manufacturing these fibres soon. Tetoron, the Japanese equivalent to Terylene or Dacron, should be in commercial production this year under licence from Imperial Chemical Industries Ltd. The highly absorbent synthetic fibre invented by Dr. Tomonari is being produced in quantity (32 million pounds in 1957, an increase of eight million pounds).

The following details of Japanese output of chemical fibres is given (in million pounds):

	1957	1956
Rayon filament ... ..	205	181
High tenacity rayon (mostly for tyres) ...	34	22
Rayon staple fibre ... ..	692	682
Acetate (mainly filament) ... ..	14	13
Cuprammonium (mainly filament) ... ..	21	19
Nylon (including Perlon type) ... ..	49	34
Vinylon (mostly staple) ... ..	32	24
Saran-type (Vinylidene) ... ..	7	5
Polyvinyl chloride ... ..	4	1
Acrylics (Orlon, Acrilan type) ... ..	1	1
Tetoron polyester ... ..	•	•

\* Less than 1 million lb.

1,059 981

Cuprammonium spinning, at present rather out of favour in many countries, is being continued in Japan. Another noticeable point concerning spinning techniques is that Toho Rayon Co. Ltd. have developed a process for making acetate by acetylating viscose rayon. The spinning is carried out from an aqueous instead of from a solvent solution.

The above and further information is given in the 1958 Japan Chemical Fibres Association publication, 'Rayon and Synthetic Fibres in Japan'.

## OEEC Report on W. Europe's Petrochemical Industry

THE POST-WAR investment in Western Europe's petrochemical industry reached \$550 million by the end of 1957, an increase of nearly \$140 million since the end of 1956. The total is expected to rise even more rapidly to \$1,200 million by the end of 1960. These figures are given in the OEEC annual review of the petrochemical, plastics materials and dyestuffs industries, by the petrochemical working party under the chairmanship of Prof. G. Robesti, of Italy.

Total carbon content of petrochemicals produced in member countries rose from 470,000 tons in 1956 to 630,000 tons in 1957, and the 1958 figure is expected to reach 830,000 tons. By the end of 1960, if investment continues as planned, a total production of over 2 million tons is forecast. This expansion will include synthetic rubber of various types, polythene and ethylene oxide derivatives, and solvents and raw materials for plastics and detergents.

The plastics materials working party (chairman W. Mauss, of Germany) reports that in 1957 total sales of plastics materials in OEEC countries reached the record figure of 1.4 million tons—19 per cent more than in 1956 and over four times the figure for 1950. Most rapid expansion was again in the thermoplastics group, where sales rose by 24 per cent to reach nearly 680,000 tons. Production of thermosetting plastics rose by 17 per cent in 1957 to 570,000 tons.

Although a fall in consumption during 1958 is forecast by the dyestuffs working party (chairman Mr. Francoise Brichet, of Switzerland) the output of dyestuffs in the OEEC countries in 1957 also reached a record figure—124,000 tons, 11 per cent higher than in 1956. The downward tendency of prices was checked.

## Physical Methods in High Polymer Research

A one-week residential course on physical methods in high polymer research is to be held at the National College of Rubber Technology, Holloway Road, London N7, from 25 to 29 August.

## Holmes Benzole Storage Tanks for E. Midlands Gas Board Plant



Three benzole storage tanks are shown being dispatched by W. C. Holmes and Co. Ltd. to the East Midlands Gas Board Grimsby Works, Sheffield. The tanks are part of the benzole recovery plant, which will have a gas capacity of 7 m. cu. ft. per day and a recovery of 80 per cent, being installed there

# FLUORINE CHEMISTRY SYMPOSIUM-2

## Consideration of Inorganic Fluorinating Agents

FLUORINE could be introduced into organic molecules by a wide variety of fluorinating agents, stated M. W. Buxton, Imperial Smelting Corporation Ltd., in his paper given at the 'Symposium on organic fluorine chemistry' held at the Central College of Further Education, Carlett Park, Eastham, Cheshire, recently (see also CHEMICAL AGE, last week).

Choice of fluorinating agent depended on the compound to be fluorinated, but in general, these agents could be divided into two broad groups:

### Group A

#### Reagents based on fluorine

Fluorine gas

$\text{AgF}_2$ ,  $\text{CoF}_3$ ,  $\text{MnF}_3$ ,  $\text{CeF}_4$

Halogen fluorides

$\text{ClF}_3$ ,  $\text{BrF}_3$ ,  $\text{IF}_5$

### Group B

#### Reagents based on hydrogen fluoride

$\text{HF}$ ,  $\text{SbF}_3$ ,  $\text{SbF}_5$ ,  $\text{HgF}$ ,  $\text{AgF}$ ,

$\text{KF}$ ,  $\text{CrF}_3$ ,  $\text{FeF}_3$ ,  $\text{HgF}_2$ ,  $\text{PbF}_4$

#### Fluorine as a fluorinating agent.

Smooth fluorination has been achieved by the following methods: vapour phase fluorination; liquid phase fluorination; fluorination with higher metallic fluorides; and halogen fluorides.

Vapour phase fluorination had been further developed by Bigelow *et al.* The newer method uses a jet fluorination reactor, in which metal packing is dispensed with. Reaction occurs in an unpacked space essentially without flame and at relatively low temperature. Cracking is thus minimised and the products are less complex mixtures than were obtained using the earlier method.

Liquid phase fluorination had not been used to any extent, Buxton stated. The method was restricted, because of the difficulty of finding a suitable inert solvent which will absorb fluorine, the compound to be fluorinated, and also the hydrogen fluoride produced. Pyridine appeared to be a suitable solvent especially at low temperatures ( $-40^\circ\text{C}$ ).

A recent method for fluorinating a liquid hydrocarbon was described in which no inert solvent was used. A pre-cooled mixture of nitrogen and hydrogen is bubbled through the hydrocarbon in a cooled glass vessel, illuminated with visible or ultra-violet light. The reaction is mainly at the gas/liquid interface as little fluorine dissolves in the hydrocarbon. Efficient stirring is required to dissipate the heat evolved. Exhaust gases are led off over  $\text{NaF}$  and condensed. As fluorination proceeds, the molecule becomes more resistant to fluorination, even under illumination.

Fluorinating agents such as the higher metallic fluorides ( $\text{AgF}_2$ ,  $\text{CuF}_3$ ,  $\text{MnF}_3$  and  $\text{CeF}_4$ ) replaced hydrogen and halo-

gen atoms by fluorine. The process was often called the Fowler Process. Important variables were: reaction temperature, hydrocarbon input rate and degree of exhaustion of  $\text{CoF}_3$ . A disadvantage of the process was that half the available fluorine was wasted as  $\text{HF}$ . Recent work by the Birmingham school had shown that it was possible to modify the reaction conditions to give a range of partially fluorinated products.

One of the advantages of the halogen fluorides over fluorine was that they were 'on tap' in quantity and independent of electrolytic cells. Also liquid phase reaction could be carried out at relatively low temperatures and were easy to control and operate.  $\text{ClF}_3$  or  $\text{ClF}$ , diluted with nitrogen was passed through a well stirred charge of the material to be fluorinated. Product is washed with aqueous  $\text{NaOH}$ , then with water and dried. Unreacted  $\text{ClF}$  is removed by scrubbing with  $\text{NaCl}$  and  $\text{CaCl}_2$ .

A similar technique was used for bromine fluorides except these were vapourised in a boiler.

### Use of Hydrogen Fluoride

In group B, hydrogen fluoride was widely used as a fluorinating agent both by itself and in combination with other reagents. Fluorocarbons and derivatives of these could be produced without the necessity of generating fluorine using an electrochemical process due to Simons which was now in pilot plant scale operation by the 3M's Co., US.

Addition of hydrogen fluoride to unsaturated bonds was a useful method for synthesising partially fluorinated compounds. A convenient method was to bubble a mixture of hydrogen fluoride and acetylene through fluosulphonic acid at  $0^\circ\text{C}$ , when smooth addition occurs to give 1:1-difluoroethane. Homologues of acetylene yielded 2:2-difluorides exclusively. Non-catalytic addition of  $\text{HF}$  to olefines went with ease in the liquid phase, the olefine being introduced into liquid  $\text{HF}$ , under pressure if necessary, at low temperatures, with stirring. Vinylic halogens on both sides of the double bond impeded and might entirely prevent addition. Addition was possible, however, in the presence of a catalyst.

Two fluorine atoms could be added across a double bond by means of lead tetrafluoride prepared *in situ* by action of hydrogen fluoride on lead dioxide. Hydrogen fluoride could also be used for introducing fluorine atoms into the aromatic ring by the Balz-Schiemann diazo reaction. A wide range of partly fluorinated derivatives had been pro-

duced by this reaction e.g., mono, di, tri and tetra-fluorobenzenes, fluoro derivatives of naphthalene, nitrobenzene, benzoic acid, phenols, etc.

*Fluorination by means of exchange reactions.* Any of the group B reagents would bring about replacement of halide in organic compounds by fluorine. Organic iodides were more reactive than bromides, which were more reactive than chlorides. However, because of cheapness, chlorides were more often used. All replacements had to be carried out under anhydrous conditions. Glass equipment could be used for easy replacements, steel was suitable, and platinum, nickel, copper and magnesium had been used. It was advantageous to carry out the reaction in an autoclave surmounted by a fractionating column and release needle valve, so that the desired fluorides were removed continuously as they were formed.

*Hydrogen fluoride reactions.* Exchange with hydrogen fluoride only occurred with very reactive halides or with compounds containing the  $-\text{CX}_3$  grouping. Such reactions generally required high temperature and pressure.

*Antimony halides.* When antimony trifluoride was used as a fluorinating agent, small amounts of pentavalent antimony salts were necessary. Antimony trifluoride did not attack alkyl halides. A  $-\text{CCl}_3$  group attached to an adjacent double bond, or to a benzene ring, exchanged readily. A  $-\text{CCl}_3$  group attached to a hydrocarbon chain was more resistant to fluorination and was usually converted only to  $-\text{CClF}_2$ .

Industrially, antimony salts were usually used together with hydrogen fluoride as a source of fluorine. The reactions were carried out under pressure at elevated temperature. This process had been applied to fluorination of carbon tetrachloride, chloroform and hexafluoroethane. The aliphatic chlorofluorides so formed were known as Freons in the US and as Arctons and Isceons in this country. Another method of preparing these was the use of hydrogen fluoride alone over carbon catalysts carrying ferric fluoride or over chromium fluoride at  $300$  to  $450^\circ\text{C}$ .

*Other fluorides.* Potassium fluoride was a mild agent useful in the preparation of mono-fluoro compounds from corresponding chloro-compounds. Silver and mercury fluorides were capable of effecting all the replacements which can be accomplished by other inorganic fluorides, and in addition some replacements which could not be done with others, e.g., alkyl halides were transformed by silver fluoride into alkyl fluorides. However, as silver fluoride was deliquescent and difficult to handle, mercuric and mercurous fluorides were more convenient reagents. Mercuric fluoride produced *in situ* by reaction of hydrogen fluoride with mercuric oxide was one of the most active fluorinating agents, readily converting alkyl halides and polyhalides to fluorides.

# DISTILLATES

★ UNEXPECTED arrival of a party of 34 Soviet scientists at the opening meeting of the international high-polymer conference at Nottingham last week caused some concern. The meeting hall was already full to capacity with 500 conference members, including an official delegation of 12 Russians and nearly 150 other foreign visitors.

The fact that an extra 34 Russians flew from the Soviet Union to attend this conference, one of the most important to be held on high polymers in recent years, is not surprising in view of the fact that Mr. K. has more than once stated that the USSR must urgently develop her plastics and synthetic fibre industries.

The high quality of the papers presented is clearly indicated by the special reports carried by CHEMICAL AGE. The first of these appeared last week, while further papers are summarised this week in pages 189-192.

★ TO GAIN a fair share of Canada's £5,000 million import market, British industry must initiate a campaign of aggressive selling and publicity. So says Sir William Rootes, who led the Dollar Exports Council delegation to Canada last spring. Speaking in London last week he added that with the fantastic growth rate of Canadian industry and population the sky was the limit. The Council is 'to use all the usual public relations channels and call meetings of industrialists'.

When I asked Sir Norman Kipping, F.B.I. director-general, what the delegation would say to the chemical industry he told me that there were more than 300 different UK industries, and that 'We cannot particularise; it is up to the chemical industry to take note of our report and act accordingly'.

This hardly ties up with Sir William's promise of an aggressive selling campaign. It may be that having issued its very broad survey, 'A report to British industry', the delegation regards its work as largely over. Surely, however, it should mark the beginning of an intensive PR drive to the UK industries mainly involved. The chemical industry is one that has much to gain from the Canadian market; Canada now imports from the US chemicals worth \$250 million a year; from the UK, only £23 million worth.

The Canadian Government publishes a detailed list of chemical and other imports, with countries of origin and dollar values. Mr. H. Watson Jamer, Agent-General in London for the Atlantic Provinces, advised me to get a copy from Canada House, London. Always ready to rally round the cause of 'aggressive

selling', I applied for a copy, to find that only two exist at Canada House; one on the files and the other 'out on loan'. Thanks to a commendable service the loaned copy was promptly recovered and was in my hands within 24 hours.

★ ASHBURTON CHEMICALS and their parent company, Geigy, must be wishing they had selected another site for their new plant than the one at Glynllifon, Caernarvon. When this project was first mooted, there was, despite a minority outcry from those who thought the plant would despoil the amenities, general approval that more employment was being brought to North Wales.

Since then, however, the project has been clouded by further controversy. The Labour Party's allegation that Geigy had been officially warned that a future Labour Government might nationalise the new plant was a storm in a teacup compared with the water dispute.

The Gwynedd River Board and the County Council are currently embroiled in an argument as to where a reservoir for the chemical works should be built. Caernarvonshire CC want a new reservoir. The board engineer wants to raise the level of Nantlle Lake, which would save ratepayers' money. I imagine that all Geigy want is a plant with as cheap a water supply as possible.

Now the Welsh Board for Industry has stepped into the picture and expressed concern at the seemingly interminable delays over the scheme. It alleges that this is being caused by the area river board, which it appears has decided to engage its own industrial water consultant.

★ MUCH interest was aroused by the announcement some months ago of the availability of p.t.f.e. dispersion coating facilities at the Tottenham Factory of Siemens Edison Swan Ltd. This coating can be applied to articles of almost any size and newly installed plant includes ovens capable of accepting parts considerably bigger than a motor car, while conveyor ovens will handle large quantities of small parts.

New applications for this process are being discovered daily and at present include the surfacing of rollers, moulds, hoppers, etc., used in the chemical, plastics, textile, papermaking and other industries, while the company has been called upon recently to coat the inside surface of xanthating churns, used in textile manufacture.

The thickness of the coating varies according to the needs of the article being

coated, i.e. resistance to abrasion, etc., but usually ranges from about .0005 to .004 in. Coatings of this order stand up to wear remarkably well and, although p.t.f.e. is still comparatively expensive, the thin deposit which is usually adequate for most purposes makes treatment of quite large articles an economical proposition.

★ MINISTRY recommendations issued recently on the safe use of fluoroacetamide as an insecticide bear out the findings of Dr. M. A. Phillips (see CHEMICAL AGE, 19 July, p. 104). The Ministry recommend that when in aqueous formulation containing not more than 1 per cent active ingredient, fluoroacetamide need not be included in the Agriculture (Poisonous Substances) Regulations. When formulated at a greater strength, the material becomes a 'second schedule part II substance'.

In view of the processing undergone by the crop, the Ministry considers that no hazard would arise from the consumption of refined sugar, molasses and spent pulp obtained from sugar beet treated with fluoroacetamide. Dr. Phillips suggested a forbidden period of 4 to 6 weeks; the Ministry say that provided not more than two applications totalling 6 oz of active ingredient per acre are made, the last not less than five weeks before harvesting, there would be no hazard to animals which eat the treated beet tops.

The MAFF feel that there is not enough evidence on which to base recommendations for other edible crops. It is stressed that animals and poultry should be kept away from sprayed areas for at least two weeks; of course, great care must be taken not to contaminate neighbouring crops, ponds, streams, rivers and water courses.

★ PRODUCTION of lithium and its salts by Associated Lead Manufacturers Ltd. at Bootle, Liverpool, has now reached the commercial stage and this has necessitated the installation of more evaporators for handling the liquors involved.

Two Kestner evaporators have been working satisfactorily for two years at Bootle, a third is now being erected and a fourth is on order. The first, with stainless steel contact throughout, is working on lithium sulphate and is a single effect double circulating climbing film evaporator under vacuum. The second is a double effect climbing and falling film non-vacuum evaporator with mild steel contact working on lithium hydroxide.

The third is a forced circulation salting type which will work non-vacuum with twin calandrias so connected that either may be isolated and cleaned while the plant is running. The fourth, another double effect Kestner evaporator, will be a duplicate of the second and will be used for concentrating lithium sulphate.

*Alembic*

# HIGH POLYMER CONFERENCE—2

## Heterogeneous Copolymerisation of Methacrylate Esters

Quite a promising field of investigation was seen in the heterogeneous copolymerisation of small quantities of water-soluble monomers with larger quantities of water-insoluble monomers. R. J. Parsons and H. Warson (Vinyl Products Ltd., Carshalton, Surrey) have therefore undertaken investigations to elucidate the copolymerisation of methyl methacrylate and sodium methacrylate.

It was known from the patent literature that many water-soluble salts of copolymerisable acids could be copolymerised with water in soluble, polymerisable esters when the latter were dispersed as an aqueous emulsion. Parsons and Warson have studied copolymerisation of methacrylate esters, especially methyl methacrylate, with sodium methacrylate, the former being maintained in a fine particle size with the aid of a water-soluble colloid and rapid stirring. Results have been reproducible. Analytical methods have been worked out for each monomer in the presence of the other, in order to follow the course of the reaction. Several ratios of methyl methacrylate and sodium methacrylate have been investigated under semi-adiabatic conditions, using a persulphate as polymerisation initiator. When the two monomers were polymerised together in equimolecular ratios, there was a period of inhibition or retardation only in respect of the sodium methacrylate, but this could be avoided by adding methyl methacrylate to sodium methacrylate at incipient polymerisation of the latter.

It had been possible to deduce a general picture of polymerisation, which was as follows: the polymerisation of methyl methacrylate followed zero-order kinetics for a large part of the reaction, while that of sodium methacrylate followed first-order kinetics. On increased dilution of the system, the rate of polymerisation of methyl methacrylate was increased. On the one hand, increase of methyl methacrylate keeping water and sodium methacrylate constant resulted in little difference in the rate of polymerisation of sodium methacrylate or on the proportional rate of polymerisation of methyl methacrylate. On the other hand, the actual rate of polymerisation of methyl methacrylate was doubled.  $\beta$ -Ethoxy ethyl methacrylate and ethyl methacrylate copolymerised to a lesser extent with sodium methacrylate in the examined ratios. In these cases a larger proportion of the sodium methacrylate remained unpolymerised when polymerisation of the ester was virtually complete. Copolymerisation was virtually nil between butyl and sodium methacrylates.

The possible mechanism of the copoly-

L. to r. Dr. P. J. Flory, Mellon Institute, US; Sir Harry Melville, conference chairman; Professor G. Gee, Manchester University; and Professor S. Medvedev, Moscow



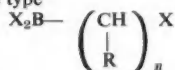
merisation was discussed. In the systems where addition of methyl methacrylate had been delayed, a copolymer was formed at the start which would not be soluble in the discontinuous phase. It would, however, assist copolymerisation by acting as an emulsifying agent in its own right, thus bringing the particle size of the methacrylate monomer to the order of 1 micron. The fine particle size emulsion was formed only in the second stage of the first system, the investigators reported.

It was suggested that there was a tendency

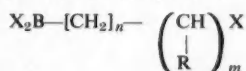
toward alternation of the two monomers. The methyl methacrylate radical  $-\text{CH}_2\text{C}(\text{CH}_3)\text{COO}-$  tended to add on the methacrylate ion  $(\text{CH}_2:\text{C}(\text{CH}_3)\text{COO})^-$ , rather than a molecule of methyl methacrylate, as this would require less energy owing to resonance stabilisation of the methacrylate ion radical. Because of the tendency of like charges on the ions to repel each other, the methacrylate ion radical  $\text{CH}_2\text{C}(\text{CH}_3)\text{COO}^-$  would tend to add on methyl methacrylate monomer in preference to another methacrylate ion.

## Polymerisation of Diazoalkanes Catalysed by Boron Compounds

A mechanism was proposed for the polymerisation of aliphatic diazo compounds catalysed by boron trifluoride and similar boron derivatives which satisfactorily accounts for the observed kinetic and molecular weight results by Professor C. E. H. Bawn, A. Ledwith and P. Matthies (Department of Inorganic and Physical Chemistry, University of Liverpool). The final polymer molecule was shown to be a boron alkyl of the type



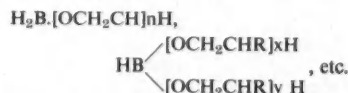
Such molecules were catalysts for the polymerisation of fresh diazo compounds and in this manner it had been possible to prepare block copolymers of the type



The mechanism of polymerisation was shown by these workers to be similar to the polymerisation of ethylene catalysed by aluminium alkyls and possibly to the Ziegler-type polymerisation of  $\alpha$ -olefins.

Thus the mechanism proposed for the polymerisation of diazoalkanes was suggested as involving co-ordination with the boron catalyst followed by migration of a group originally attached to the boron atom. The net result was a series of insertions of monomeric units into a boron-alkyl bond.

Bawn *et al.* reported that similar behaviour involving boron had been observed in the reactions between diborane and aldehydes and ketones in which case the reaction stopped after the first insertion, while alkylene oxides reacted with diborane to give products of the type



L. to r. Dr. L. Breitman, Polymer Corporation, Ontario; Dr. H. Ende, Mainz University; and R. S. Colborne, a consulting chemist from Chislehurst

The above reactions are closely analogous to the polymerisation of olefins catalysed by aluminium alkyls, since both reactions involve the stepwise anionic growth of a metal-alkyl chain.

Since diazomethane is much more reactive (nucleophilic) than ethylene it is able to react readily with boron alkyls whereas ethylene does not. Aluminium

alkyls are more electrophilic than the corresponding boron alkyls and, as Ziegler has shown, they will react with olefins at slightly elevated temperatures and pressures.

These workers indicated that it was to be expected, therefore, that aluminium alkyls react even more readily with diazoalkanes, and this and related reactions were now being investigated.

## Graft Copolymers from Natural Rubber Latex

**G**RAFT copolymers have been prepared from natural rubber latex and methyl methacrylate using visible, ultra-violet, and  $\gamma$ -radiation by W. Cooper, G. Vaughan, S. Miller and M. Fielden (Dunlop Research Centre, Birmingham). The influence of monomer concentration, temperature, radiation intensity, and, in the case of photoinitiation, photosensitiser concentration, on polymerisation rates and molecular weights was studied. The proportion of graft polymer is stated to be very high initially but falls with conversion and with increase in monomer concentration.

Photoinitiated grafting was found to depend on the photosensitiser and on temperature, but with  $\gamma$ -initiation the fraction grafted was independent of temperature. In the latter case the high proportion of graft could not be explained in terms of relative  $G$  values and con-

centrations of monomer and rubber.

The fall-off in proportion of graft with conversion was thought to result from microscopic heterogeneous regions formed during polymerisation, which were believed to increase in size as polymerisation proceeded, thereby reducing further the possibility of grafting. Degradation of previously grafted polymers by prolonged irradiation was not of consequence in this case.

Molecular weights of grafted and ungrafted polymers were very similar, except at low monomer concentrations, and a marked feature of this system was their constancy with change in all reaction conditions other than monomer concentration. Estimates of the utilisation of radiation energy have been made and these workers concluded that on an energy basis  $\gamma$  and photoinitiated reactions were of comparable efficiency.

## Aluminium Alkyls as Catalyst for Polymerising Propylene Oxide

**T**HE utility of several alkyl aluminium compounds as catalysts has been investigated by Philip E. Ehrt and Charles C. Price (Department of Chemistry, University of Pennsylvania, Philadelphia, US). Polymerisations were carried out under a dry nitrogen atmosphere in sealed Pyrex tubes. Effective catalysts were triethyl aluminium, diethyl aluminium chloride and triisobutyl aluminium, and small but reproducible yields of isotactic polymer were obtained.

It is reported that the aluminium alkyl systems were optically clear to the naked eye and showed no evidence of precipitation before or during polymerisation.

## New Catalysts for Preparing Isotactic Polypropylene Oxide

**I**N the paper by Maseh Osgan and Charles C. Price (Department of Chemistry, University of Pennsylvania, Philadelphia, US), on 'New catalysts for the preparation of isotactic polypropylene oxide', an extension of earlier work by the authors on the Pruitt-Baggett catalyst system (i.e., modified ferric catalysts) is reported. They have found that a wide variety of Lewis acid metal alkoxides, or mixtures of alkoxides and halides, serve as catalysts for the stereospecific polymerisation of propylene oxide, some at rates considerably more rapid than the Pruitt-Baggett catalyst.

An alternative procedure has been employed to prepare the second catalyst system. One mole of propylene oxide was added to ferrous (rather than ferric) chloride in ether and then chlorine added to oxidise the ferrous ion to ferric. This catalyst is described as a white powder which gave somewhat faster polymerisation.

'Autoinhibition' noted with aluminium isopropoxide was overcome by adding anhydrous zinc chloride, when rapid and complete conversion to polymer occurred. Aluminium chloride was not as effective as a cocatalyst as zinc oxide. The atactic polymer from these cocatalyst systems is stated to be of much lower molecular weight ( $(\eta)_{0.2-0.4}$ ) than the isotactic polymer.

## Isotactic Polymerisation of Phenyl Glycidyl Ether

The effect of catalysts on the polymerisation of monomers related to propylene oxide has been investigated by Allen Onshay and Charles C. Price (Department of Chemistry, University of Pennsylvania, Philadelphia, US). In this instance polymerisation of phenyl glycidyl ether (phenoxy propylene oxide) is reported.

Phenyl glycidyl ether has been polymerised, using aluminium catalysts, to crystalline as well as amorphous polymers. The crystalline fraction was high melting (ca 200°C) and insoluble at room temperature. The amorphous fractions were soluble at room temperature and some showed rubber-like properties. Best yields of the polymer were obtained, it is stated, when reactions were carried out in bulk, in open, stirred systems, using aluminium isopropoxide and zinc chloride as cocatalysts.

Investigation of the physical properties of this new isotactic polymer is being actively pursued by these workers.

The full papers will be published by Interscience Publishers Inc., US.

### Change of Name

Shell Chemicals Distributing Co. of Africa Ltd., St. Helen's Court, Great St. Helens, London EC3, have changed their name to Shell Chemical Co. of Africa Ltd.



At the reception, l. to r.: Dr. R. L. Vale, UKAEA, Wantage Radiation Laboratories; Dr. R. S. Lehrle, chemistry department, Birmingham University; Dr. F. L. Dalton, Wantage; Dr. P. R. Sewell, Wantage; and Dr. P. E. M. Allen, Birmingham University

## Grafting of Polycaprolactam on a Polymethacrylate-Type Backbone

INVESTIGATIONS by O. Wichterle and V. Gregor (Technical University, Prague, Czechoslovakia) have shown that  $\epsilon$ -caprolactam could be very readily polymerised by alkaline catalysts even at room temperature provided diacylamines, such as *N*-acetyl caprolactam, were present. As part of the investigation of the mechanism of this reaction, these investigators have prepared polymers of *N*-methacryloyl caprolactam containing the active secondary imide groupings attached to a polymethacrylate-type chain.

Methacryloyl caprolactam itself would not, it is reported, for obvious steric reasons, undergo polymerisation, but its copolymers could readily be obtained; thus it would form a copolymer with styrene with the parameters  $r_1=1$ ,  $r_2=0$ . When this copolymer is allowed to react with caprolactam in the presence of alkaline catalysts, the monomer can enter at the sites of imide groupings. Simultaneously with the addition of the lactam to the copolymer, crosslinking of the polymer takes place which causes the formation of insoluble gels at the very beginning of the grafting.

The assumption is made that crosslinking is due to a reaction of the Claisen condensation type between two diacyl imide groups. This is supported by the fact that the relationship of the reciprocal specific viscosity and time is approximately linear in solution polymerisation. The grafting of lactam which is not controlled by diffusion continues at an undiminished rate even after the gel formation. The rates of grafting and crosslinking are proportional to the concentration of sodium caprolactam added.

## Preparation of Block Polymers by Mechanical Shear

PREPARATION of block polymers by mechanical shear of polymer-monomer systems was discussed by D. J. Angier, R. J. Ceresa and W. F. Watson (The British Rubber Producers' Research Association, Welwyn Garden City, Herts.) The mastication of polymers brought into a rubbery state by admixture with monomers caused polymerisation of the monomer. The reaction showed dependence on rate of shearing, temperature, and monomer concentration expected for a mechanism of mechanical rupture of the polymer to free radicals which initiated polymerisation.

Fractional precipitation methods for separating block polymers of polymethyl methacrylate-polystyrene from the homopolymers were applied to the products after masticating both initial polymer-monomer combinations. The products of reaction indicated that the block polymer did not contain many segments of each polymer. When the two polymers were present either as free or block polymers, the polymethyl methacrylate was preferentially ruptured to form initiation sites.



L. to r. Dr. R. Hill, research director, ICI Fibres Division; Sir Harry Melville, conference chairman; Professor G. Natta, Milan; Professor F. S. Dainton, Leeds University; and Dr. R. F. Boyer, Dow Chemical Co, Michigan

## Polymerisation of Epoxides by Metal Halide Catalysts

AS polymerisation of ethylene and propylene oxides was well known but had not been studied in great detail, and as extension to other epoxides offered an interesting field of work (both as a study in a relatively untouched branch of polymerisation chemistry, and for the possibilities of new polymers which might be expected), R. O. Colclough, G. Gee, W. C. E. Higginson, J. B. Jackson and Morton Litt (Department of Chemistry, The University of Manchester) commenced investigating both aspects systematically. A preliminary study has been made of the polymerisation of a number of epoxides by basic catalysts. The reactions did not readily lead to high molecular weight products and other types of catalyst, including a range of metal halides, have been studied.

Observations made by C. L. Price and M. Osgan (*J. Am. Chem. Soc.*, 1956, **78**, 4787) have been repeated, paying particular attention to the role of water, which has been found by these Manchester workers to exercise a marked degree of control over the course of the reaction. It is reported that a fairly broad survey of metal halides has shown that ferric chloride is by no means unique in its behaviour and that these catalysts can also be applied to other epoxides.

No detailed study of the polymers obtained has been made as yet. Melting points, where recorded, have been measured with the aid of a Koeffler hot plate and polarising microscope using a rate of heating of approximately 2°C/minute. Intrinsic viscosities were measured in benzene solution at 25°C. A few preliminary osmotic pressure measurements have been made in Zimm-Meyerson osmometers.

Halides investigated for ethylene oxide polymerisation were found to fall into three main groups: those soluble in monomer and catalytically active ( $AlCl_3$ ,

$SbCl_3$ ,  $BeCl_2$ ,  $BCl_3$ ,  $FeBr_2$  (? mixture of  $FeBr_2$  and  $FeBr_3$ ),  $FeCl_3$ ,  $SnCl_4$ ,  $TiCl_4$ ,  $ZnCl_2$ ); soluble but inactive ( $AsCl_3$ ,  $SbCl_5$ ,  $CoCl_2$ ,  $CuCl_2$ ,  $FeCl_2$ ,  $HgCl_2$ ,  $NiBr_2$ ,  $NiCl_2$ ,  $ZrCl_4$ ); insoluble and inactive ( $CdCl_2$ ,  $CuCl$ ,  $MgCl_2$ ,  $HgCl$ ). All experiments giving good yields of polymer gave products of similar intrinsic viscosity, indicating molecular weights of the order of 10,000, it was stated. These were described as all brittle, waxy, crystalline materials melting in the range 56-60°C. They did not appear to differ in any important way from commercial polymers having estimated molecular weights of a few thousand.

With  $FeBr_2$  (?) tougher products were obtained, of higher intrinsic viscosity, melting above 60°C and which were of higher molecular weight (osmotic pressure of one gave  $M = 37,000$ ). A similar range of halides was investigated for propylene oxide polymerisation. The products obtained were 'largely or completely amorphous'. Production of crystalline polymer was associated with the presence of water in the reaction mixture. The amorphous polymers varied in mechanical properties from viscous liquids to rubbery solids.

Polymerisation of styrene oxide was produced using  $AlCl_3$ ,  $SbCl_5$ ,  $BeCl_2$ ,  $FeCl_3$ ,  $SuCl_4$ ,  $TiCl_4$ ,  $ZnCl_2$  and  $ZrCl_4$ . Small yields were obtained of a crystalline product (>10 per cent) melting at 125°C, which was believed to be a cyclic dimer; this was easily extracted from the reaction mixture. The polymer remaining was generally a 'highly viscous liquid' at room temperature.

Of the polymerisation mechanism, Colclough *et al.*, stated that the initial reaction featured a cationic polymerisation and it was suggested that this was its mechanism, the chain propagation being terminated by a Cl transfer. A mechanism of 'co-ordinate complex polymerisation' in which molecules of monomer first complex with the cata-

lyst  $\text{ClFe(OR)}$ , had been suggested by Price. The overall result was, considered the Manchester investigators, that a molecule of monomer was interpolated between the metal and one of the chains attached to it, and that it was clear that the process can be repeated indefinitely so long as no side reaction occurred to displace the chain from the metal. Detailed formulation of the mechanism required further study, it was stated. Key to the process was seen in the ability of the catalyst to form co-ordination compounds by acting as Lewis acid. 'Since the first step involves displacement of halogen to form Metal-OR bonds, there would seem to be no

obvious advantage in using halides in the first place.' Metal alkoxides (e.g.,  $\text{Al(OiPr)}_3$ ) were catalytically active, and this type of compound was now being studied.

A marked apparent difference in the behaviour toward ethylene and styrene oxides was noted. Catalysts  $\text{SbCl}_5$ ,  $\text{AlCl}_3$ ,  $\text{FeCl}_3$  and  $\text{ZrCl}_4$  all appeared to be of comparable efficiency towards styrene oxide, whereas toward ethylene oxide there was a sharp gradation:  $\text{SbCl}_5 > \text{FeCl}_3 > \text{AlCl}_3 > \text{ZrCl}_4$ . This discrepancy, it was suggested, was perhaps a further reflection of a difference of polymerisation mechanism between the two monomers.

## Polymethylenes and the Structure of Polythene

A STUDY was undertaken of short-main branching, its nature and effects, by A. H. Willbourn (Research Department, Plastics Division, Imperial Chemical Industries Ltd.) since the evidence on these matters was somewhat conflicting. The approach employed was the use of 'models' for polythene, such as polymethylenes, containing known numbers of branches of known length, to develop and calibrate infra-red and other techniques for subsequent use on polythene itself.

The polymethylenes used for calibration work contained deuterated butyl branches and the numbers of branches present in the polymers were established by two independent methods; direct assay for deuterium and mass spectrometric analysis of the irradiation products. Using this series a new infrared technique had been calibrated absolutely to measure total numbers of methyl groups and also butyl branches specifically.

The infrared technique was a compensation method, using solid specimens in a double-beam spectrometer, in which the compensating material (linear polymethylene) was devoid of the structural features, namely branches, to be measured. Using the same technique and reference samples of ethyl-branched polymers (Hydropol), a method of estimating ethyl branch concentration was developed.

A study of the gaseous products of irradiation of the butyl-branched poly-

methylenes, using mass spectrometric analysis, had shown the extent to which this technique might provide a quantitative means of identifying and estimating branches in such polymers; it had also greatly strengthened the evidence of earlier workers that low density polythenes contained primarily ethyl and butyl branches.

The same conclusion, in more quantitative terms, was drawn from an infrared study of various polythene specimens: practically all the branches observed could be accounted for as ethyls and butyls present in the ratio of approximately 2:1. Some high density polythenes contained a few branches, which appeared to be ethyl groups. The effects of the numbers and lengths of branches on degree of crystallinity, crystalline melting point, density, rate of oxidation, and dynamic mechanical properties were illustrated and discussed by Mr. Willbourn.

Regarding crystallinity and density, it was reported that a survey of the behaviour of these polymers indicated that their molecular weights were so large that once the polymers have been melted their crystallinities depended largely on their subsequent thermal history, and that it was practically impossible to return to the original crystallinity values even after very careful annealing treatment.

Two linear polymethylenes (P.2 and P.5) had densities of 0.97 and 0.98 and were more than 90 per cent crystalline;

values of both properties fell on moulding and were not regained even on careful annealing. Marlex 50 sample was annealed to give a density of 0.979 and 90 per cent crystallinity, but the melt viscosity of this polymer was  $2 \times 10^6$  poise. Because of these complications, Willbourn stated that it was impossible to find a correlation between degree of branching as such and either density or crystallinity.

Melting points of various polymethylenes were examined. Certain linear polymers (P13 and P15) had maximum values of  $136^\circ$  and  $138^\circ\text{C}$  respectively. Melting ranges of the branched polymers were found to be unexpectedly large and not very reproducible, possibly due to oxidative degradation, it was suggested. For methyl branches the melting point was lowered by about  $0.6^\circ\text{C}$  per  $\text{CH}_3$  group per 1000 chain atoms. Rate of oxidation of polymethylenes seemed to depend both upon the degree of branching and on the amorphous content, it was reported.

## Aluminium Catalysts for Ethylene Oxide Polymerisation

WORK has been undertaken by Richard A. Miller and Charles C. Price (Department of Chemistry, University of Pennsylvania, Philadelphia, US) to evaluate with ethylene oxide the catalysts found suitable for preparation of isotactic polypropylene oxide. They report that both aluminium iso-propoxide-zinc chloride and the aluminium alkyl catalysts are effective in converting ethylene oxide to polymer of higher molecular weight than any previously reported. Estimates of hydroxyl and unsaturated end groups were made by a method previously described by L. E. St. Pierre and Charles C. Price (*J. Am. Chem. Soc.*, 1956, **78**, 3432) for polypropylene oxide.

Examination of the polymer formed for hydroxyl and olefin end groups, using phthalic anhydride and bromine, revealed approximately one double bond per molecule but no detectable hydroxyl groups.

## New Type Gas Plant For Partington

A NEW plant will be built at Partington, near Manchester, to develop commercially a new gas manufacturing method: the hydrogenation of oil and coal under very high pressures. This was announced by Sir Ian Horobin, Parliamentary Secretary to the Minister of Power at the opening of a £2 million gas production station built by the North-Western Gas Board at White Lund, near Morecambe.

The new plant at Partington is to follow work at the Gas Council's research centre, Solihull, into the high pressure gasification of small non-coking coals which held out a prospect of radically cheaper gas.

## Solartron Group Plans

The first stage of extensions planned by the Solartron Group Ltd. are now nearing completion at Farnborough, Hants. A section of some 50,000 sq. ft. is expected to be in production in August.



L. to r. R. E. Bridgeford Scott, Bader Co.; Dr. G. L. Fray, Shell Chemical Co.; Dr. K. J. Clark, Shell Chemical Co.; and D. M. Woodford, Scott Bader Co.

## Analytical Review

## SIMPLICITY IN ORGANIC MICROANALYSIS

ONE of the features of organic microanalysis which disturbs and perplexes the non-specialist is undoubtedly the mysterious combustion trains, the serried ranks of Kjeldahl units, the batteries of complicated 'alkoxyl and acetyl apparatus, etc., which confront him immediately he enters a microlaboratory. The situation sometimes assumes an air of bewilderment for the inquirer when he discovers that seldom do two microanalysts use exactly the same technique or even apparatus; each one seems to have his own secret recipe for success.

Generally the factor behind the complicated apparatus, the individual *savoir faire* of the operator, the carefully timed experimental schedule, etc., is the necessity to render 'inorganic' the element (or sometimes group) which is being determined. This 'decomposition' or 'mineralisation' is not usually an easy matter and is a source of considerable trouble.

## Decomposition in oxygen filled conical flask

Recently, however, there has been renewed interest in a simple decomposition method, the basic principle of which is combustion of the sample in an atmosphere of oxygen contained in a stoppered vessel which also contains an absorbing solution to remove the oxidation products. The historical development of this method is beyond the scope of this review and it will be discussed by others elsewhere (1). It is appropriate, however, to note that the credit for recent micro-development lies with Wolfgang Schöniger (2, 3).

In the 'flask' method, the compound being decomposed is wrapped in a 1 in. square of ashless filter-paper from which hangs a wick or fuse consisting of a small strip of the same paper. The paper is sandwiched between two halves of a piece of platinum gauze attached to a platinum wire which in turn is sealed into the ground-glass stopper of a conical flask (250-300 ml). The flask contains a few millilitres of the absorbing liquid (water, hydrogen peroxide, etc.) and is filled with oxygen from a cylinder. When the flask is full of oxygen, the mouth of it is covered over with the palm of one hand while the other hand holds the stopper in such a way that the fuse of the paper wrapping can be ignited in a bunsen flame and the stopper complete with wire gauze and burning wrapping can be transferred quickly into the flask.

Within the flask, which should be tightly stoppered, oxidation proceeds rapidly and usually the semimicro- (say 20 mg.) or micro- (say 5 mg.) sample is

oxidised with incandescence within 2-3 seconds. During this process, some excess pressure may be generated and most operators therefore recommend inversion of the flask to form a water seal around the stopper. The time required for absorption of the gaseous products concerned varies, but is usually complete within 10-30 minutes. When the flask has cooled down there is usually a slight negative pressure due to the combustion of the hydrogen in the compound. The risk of explosion during decomposition seems to be small.

It is difficult to envisage a simpler method of decomposition. Indeed I freely confess that when I first became aware of the technique in 1955 I was sceptical that such a devastatingly simple device could succeed so rapidly and accurately where more complicated and seemingly more painstaking methods were sometimes not too satisfactory.

The flask methods so far devised have been concerned principally with the analysis of halogens and sulphur and mixtures of the two. In 1952 the Czechoslovak workers, Mikl and Pech (4) oxidised halogen compounds as described above, but using larger vessels and samples. Most compounds contained sufficient hydrogen to yield the halogen acid which was absorbed in distilled water in the bottom of the vessel and titrated with mercuric nitrate using sodium nitroprusside as indicator. Compounds low in hydrogen were catered for by adding some paraffin before combustion. Sulphur was determined simi-



Total apparatus requirements for decomposition of organic compounds by 'flask' method

larly by titration with standard alkali following absorption in water containing 3 per cent hydrogen peroxide. Carbon dioxide was first of all eliminated by boiling the solution for two minutes before titrating. Subsequently in another paper (5), the same authors dealt with sulphur and chlorine containing compounds by (1) determination of total acidity and (2) application of the

By

Dr. T. S. West, Ph.D

In this article Dr. West reviews a recent trend in organic microanalysis wherein organic compounds are decomposed in a remarkably simple and rapid manner. Apparatus requirements are an ordinary glass stoppered conical flask with a short length of platinum wire sealed into the inner surface of the stopper. The sample is wrapped in paper, which is suspended on the platinum wire, ignited and placed into the oxygen filled flask. Oxidation is complete in 2-3 seconds. Decomposition products are determined in the absorbing solution present in the flask.

Hg(NO<sub>3</sub>)<sub>2</sub> method *vide ultra* in the titrated solution. 100 mg. samples were used.

Schöniger first developed the micro and semimicro flask methods. In 1955, he determined the halogens (2) by absorbing the decomposition products in alkaline caustic potash, applying the Vieböck method for chlorine and bromine and the Leipter method for iodine. The method was rapid (ca 20 minutes) and accurate to within  $\pm 0.3$  per cent. Sulphur (1956) was determined (3) by application of a barium chloride/EDTA procedure. Wagner (6) (1957) effected what might generally be considered an improvement on Schöniger's sulphur method by applying the Fritz and Yamamura titration (7) to determine the sulphate. This involves direct titration of the sulphate with barium perchlorate in an 80 per cent *iso* propanol-20 per cent water medium, using Thorin as indicator. No interference was observed from phosphorus, nitrogen, chlorine or fluorine present in the compound.

In 1958 several variations of the flask method have appeared. In Hungary, Erdey, Mazar and Masel (8) observed that the Schöniger method could be improved by replacing the Vieböck halogen finish by direct titration with silver nitrate using Varanium Blue B (4-methoxy-4-aminodiphenylamine hydrochloride) as adsorption indicator (pH 3-3.6) in an acetic acid medium. The titrimetric method is an interesting one, but only time can show whether it is superior to the well tried Vieböck procedure. Like Wagner, *loc cit*, these authors favoured the Fritz-Yamamura titration for sulphate. Chlorine was determined subsequently in the titrated solution after boiling out the *iso* propanol. Boetius, Gutbier and Reith (9) used the flask method for halogens and sulphur, but preferred direct titration

with barium nitrate and Alizarin Red S for determination of the latter. Chlorine was determined in the titrated solution by a Volhard procedure following adjustment of acidity till the yellow unionised form of the Alizarin Red S indicator was obtained (pH ca 4). Alicino (10) also preferred the Fritz-Yamamura titration for the determination of flask-combusted 'organic' sulphur.

American workers (14) have applied the flask method to the determination of phosphorus in organic compounds on the micro and semimicro scales. The phosphorus pentoxide was absorbed in nitric acid in the flask, boiled to convert to orthophosphate and precipitated as magnesium ammonium phosphate. Thereafter an indirect analysis of the phosphate was achieved by applying a complexometric titration with EDTA to determine the magnesium. A colorimetric

molybdenum-blue method was preferred for micro amounts of phosphorus.

There is no doubt that this extremely simple and very inexpensive method of decomposing organic compounds for elemental analysis will find favour in many quarters and many new applications can confidently be awaited.

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## Value of Buying Contract Designs By The Project

(Letters to the Editor)

SIR,—I was interested in the arguments and conclusions contained in your leading article 'Contract Research' (CA, 19 July) because they correspond almost word for word to those advanced in connection with contract designing of engineering equipment. The same three criticisms (conflict of interests—client's disclosure of confidential information—specialised knowledge) are levelled against the idea of employing design firms; the same answers which you make will equally meet those criticisms when applied to contract designing.

Fortunately, in the case of contract designing, small firms are beginning to value the advantages of buying services 'by the project', thus obtaining the optimum weight of design man-power at the right moment in the work and having the benefit of more 'fresh minds' than they could hope to maintain on their staff continuously. Those who cost their own design and drawing work realistically often have, in addition, a pleasant surprise when considering the cost of contract design work.

Here, perhaps in a small way, is a chance for streamlining industry and making our very limited technical man-power go further.

Yours, etc.,

G. WITTENBERG,  
Managing Director,

Rhoden Partners Ltd.,  
29 Park Crescent,  
London W1.

### Contract Research on Chemotherapeutic Work

SIR,—We are sure that you will not mind amending your editorial of 19 July, and that our very good friends, Fulmer Research Institute and Sondes Place Research Institute, will also not mind our calling attention to the fact that this firm also undertakes sponsored or contract research and indeed has done much work on chemotherapeutic problems and on

pesticides, the results of which have been or will be published by the courtesy of our various clients.

We also are a wholly British firm and would like to see many more British consultants taking up the challenge of sponsored contracts now being given to non-British organisations.

Yours, etc.,

M. A. PHILLIPS.

Dr. M. A. Phillips and Associates,  
9 Western Road,  
Romford.

### Polythene/Polyethylene

SIR,—It is by no means uncommon for words in English to have a pronunciation different from the spelling, e.g., colonel. In chemistry, a syllable is sometimes left out, an example being dichlorobenzene, in which the second 'o' is often omitted in speech.

Cannot the polythene/polyethylene controversy be solved by a compromise—spell the word polyethylene, and pronounce it polythene?

Yours, etc.,

J. H. GREAVES.

Younghusband, Barnes and Co. Ltd.,  
Rotherhithe, London SE16.

### UK Fertiliser Prices

Last week Mr. J. B. Godber, Joint Parliamentary Secretary to the Minister of Agriculture, Fisheries and Food, gave the following figures for fertiliser prices in the UK as at 1 July, 1958:

	Price per ton nutrient delivered (without subsidy)	Price per ton nutrient delivered (with subsidy)
	£ s. d.	£ s. d.
Sulphate of ammonia (20.8 N)	102 17 6	55 5 6
Superphosphate (18 per cent. P <sub>2</sub> O <sub>5</sub> )	80 2 0	42 12 0
Basic slag (12 per cent. P <sub>2</sub> O <sub>5</sub> )	56 5 0	31 13 4
Muriate of potash (60 per cent. K <sub>2</sub> O)	36 9 6*	36 9 6

\* Export.

## Dollar Area Chemical Imports to be Decontrolled

FROM 18 August, the import licensing restrictions will be removed from a wide range of chemicals and allied products imported from the dollar area. This decision was announced by Mr. J. K. Vaughan-Morgan, Minister of State at the Board of Trade, in a Parliamentary Answer on Tuesday. In effect, with certain exceptions such as dyestuffs and intermediates, import licences will no longer be required for most industrial chemicals.

As a result of this decision, users in Great Britain would benefit from the greater freedom in choice of supply, stated the Board of Trade. It has been possible to take this further step in the removal of discriminatory restrictions against imports from Canada, the US and other dollar areas, by virtue of the improvement in the balance of payments position.

A notice to importers, giving details, will be issued by the Board of Trade in the next few days. The open General Import Licence (No. 2) specifying the chemicals concerned is now on sale at HMSO (price 4d, by post 6d).

## Ministry Recommendations Antibiotics in Agriculture

THE Ministry of Agriculture, Fisheries and Food has issued recommendations for the safe use of the antibiotics streptomycin and oxytetracycline. The recommendations refer to the use of these chemicals on stone fruits and on crops which will not be used for human or animal consumption. Spraying streptomycin and oxytetracycline on cherries should not offer a hazard to consumers, provided the preparations are not used between petal fall and harvesting nor at a rate in excess of 3½ oz. streptomycin activity and/or ½ oz. oxytetracycline activity per 100 gal. of high volume application (or correspondingly for medium and low volume application). One per cent streptomycin emulsion paint employed after harvesting of stone-fruit trees should not prove dangerous. The Ministry state that there is insufficient information available to enable recommendations to be made regarding the safety of other crop usage.

Streptomycin and oxytetracycline have not needed to be included in the Agriculture (Poisonous Substances) Regulations, but operators are urged to wash off thoroughly with soap and water any of the concentrate which touches their skins; to wash all exposed parts of the body before eating, smoking, etc., and on completion of the operation. Fully protective clothing should be worn. Further details may be obtained from the Ministry.

## Fluorine Corrosion Data

A 24-page booklet on 'Handling Fluorine and Fluorine Compounds with Inco Nickel Alloys', has been published by The International Nickel Co. Inc., 65 Wall Street, New York 5, US. The publication summarises corrosion data and industrial experience gathered by the company from a number of sources.

## Overseas News

# CHINA'S PLANS FOR PRODUCTION OF INDUSTRIAL AND AGRICULTURAL CHEMICALS

**R**EPORTS from China indicate vigorous plans for increasing the production of industrial and agricultural chemicals, and a broad hint that the Peoples' Republic is entering the atomic energy field.

The Peking chemical plant is being enlarged, probably to provide raw materials for an atomic energy industry. By the end of the year it is scheduled to be producing reagents, rubber antioxidants, titanium, strontium, zirconium, tantalum, beryllium and molybdenum. The Yungli-Chinta works in Tientsin are also being expanded to provide an annual output of 160,000 tons each of ammonium chloride and pure soda. A new gas compressor for the Yungli works in Nanking is to boost production of liquid ammonia by 20 per cent.

Construction of a new chemical fertiliser plant at Kaifeng, Honan, has begun, and the production target is 200,000 tons each of phosphatic and nitrogenous fertilisers. Double superphosphate is to be produced at a later stage. A fluorine-free phosphate fertiliser has been produced from quartz, phosphorous and coal dust by the Shanghai Chemical Engineering Research Institute.

A small 8,000-ton nitrogenous fertiliser plant, believed to be a prototype of 36 such plants to be built this year in various parts of China, has begun production.

New products being turned out by the Shanghai Resins Plant are ion-exchange resins and heat and cold resistant silicate plastics.

## Union Carbide To Make High Purity Hydrogen

To meet metropolitan demands for high-purity hydrogen, the Linde Company, division of Union Carbide Corporation, is now operating a new plant at Linden, New Jersey. According to the president, W. M. Haile, most of the hydrogen will be used in electronics, pharmaceuticals and for food hydrogenation and processing. Linde designed and built the plant on land leased from General Aniline and Film Corporation, who will supply crude hydrogen for processing at the Linden plant.

## Soviet Chemical Industry and Natural Gas

Soviet chemical industry development plans have, it is reported, placed a considerable burden on the country's natural gas industry. Head of the Soviet Central Gas Administration, Glaugas, Mr. A. K. Kortunov, has recently outlined the steps which are being taken to

satisfy the gas requirements of the larger chemical plants.

Research is being undertaken in developing new methods for the chemical processing of natural gas. Several new gas and benzene plants are planned. Present annual output of liquid gas in the USSR was stated by Mr. Kortunov to be 60,000 tons, but a target of 4 million tons has been set for 1965. A rapid expansion for the whole gas industry is envisaged. By 1972, it is anticipated that Russia will be producing about 350,000 million cubic yards of gas annually.

## Indian Ciba Factory in Production

Production at Ciba Pharma's new factory at Bhandup (Bombay) for manufacturing chemical, pharmaceutical and cosmetic products, started recently. This is the first plant to be established by the firm in India. Some of the principal products manufactured are Elkosine (sulphasomidine) and Formocibazol, hormones and Serpasil (reserpine). Serpasil is manufactured from indigenous rauwolfia roots. The factory will also process Coramine and Enterovioform from raw materials supplied by Atul Products Ltd., Bulsar.

## U.S. Tariff Commission's Hearing on Barium Chloride Imports

Imports of barium chloride were discussed at a recent hearing of the US Tariff Commission. The hearing was part of the Commission's escape clause investigation of the imports, begun last March at the request of Barium Reduction Corporation, South Charleston, West Virginia, US. Representatives of the company informed the Commission that off-shore supplies of barium dichloride, supplied to the US market mainly by West Germany and in smaller portions by France, were taking more and more of the market. Additional protection was requested against the imports.

## New African Fertiliser Factory in Production

The £3,500,000 fertiliser factory built by African Explosives and Chemical Industries on the outskirts of Salisbury, Southern Rhodesia, is now operating. The factory is the only one of its kind on the African Continent and is now producing phosphatic fertilisers. Triple superphosphate production is also planned. Capacity for superphosphate is 170,000 tons a year, of which two-thirds can be produced in the form of

triple supers. A pyrites-burning sulphuric acid plant has also been built with an approximate yearly capacity of 70,000 tons of acid. Raw material for making sulphuric acid comes from the Iron Duke mine at Glendale, 30 miles north of Salisbury, while phosphate rock is imported from Morocco.

## Dow's Australian Plans

Reports that the Dow Chemical Co., US, are interested in establishing a factory in South Australia have been confirmed by the chairman, Mr. E. W. Bennett, who says that such a plant would produce chemicals from by-products of the new oil refinery planned by the Vacuum Oil Co., near Adelaide.

## Uranium Extraction Plant for Sweden

Sweden's Atomic Energy Company have plans in hand for a uranium extraction plant using native schist deposits from Mt. Billingen in West Sweden. Scheduled annual output planned is 120 tons of uranium from 900,000 tons of crude schist.

The plant will be known as the Ranstad Works and will cost an estimated Kr.125 million. It is expected to be completed by 1962. Preparatory work is to be started this autumn.

## Canada's Natural Gas Exports

The Bonden Royal Commission on Canadian energy resources is preparing its preliminary report, which it is hoped will be published at the end of September at the latest. This report will deal with the export of natural gas.

Mr. Diefenbaker has already announced that no action will be taken on gas export permits until the Commission's findings are available.

## Belgium Order for Beryllium

Belgian Nuclear Planning Centre has placed a record order for fabricated beryllium with the Brush Beryllium Co., Ohio, US. Valued at \$1.2 million, the order is for two beryllium cone matrices for the high-flux material testing reactor BR2, under construction at Mol. It is scheduled to be ready early in 1960.

BR2 is shown in the Euratom Commission's report as the second of 25 existing and projected research reactors to use beryllium as a moderator. The other is the French plutonium-burning homogeneous reactor, Proserpine, at Saclay, moderated with graphite and beryllium oxide, which went critical earlier this year.

## Greek Nitrate Factory

A fertiliser factory is to be built at Ptolemais in Greece, near the Bodossakis lignite mines, at a cost of some £35 million. Capacity of the nitrate plant planned is 75,000 unit tons annually, which is equal to about 350,000 tons of fertilisers. This output will more than

cover the country's current requirements. The Greek Government has chosen the Italian company Ammonia Casale and the German company Ude to construct the plant. About \$12 million is to be lent to Greece by the US Development Loan Fund to help with the cost of the project.

### Dow's Electroless Plating Coats Magnesium With Nickel

Several plating works have now been licensed by Dow Chemical, US, to use their recently developed electroless process for nickel plating magnesium.

The new process is stated to work with various metals, including steel, aluminium and copper. A special nickel solution is employed. The metal to be plated is immersed in this solution and a better nickel surface is claimed than is obtained by electroplating. The coating can be built up to any desired thickness. Of importance are stated to be bath temperature which controls the plating rate (the hotter the bath, the faster it plates) and immersion time which determines the thickness.

The nickel deposit contains 7 to 9 per cent phosphorus so that it becomes an age hardening alloy with greater hardness and better abrasion resistance. If maximum corrosion resistance is required zinc immersion plus copper strike is used.

Easy soldering of the electroless nickel is claimed, using acid-core solder. For resin-core solder, the nickel-plated part is tin-plated first and then joined with the resin core solder.

Dow consider that the new process will find useful application in the electronics industry, which normally uses electroplated magnesium parts.

### Heavy Water By $H_2S/H_2O$ Temperature Exchange Process

Production of heavy water by the  $H_2S/H_2O$  two-temperature exchange process has recently been considered by Professor Dr.-Ing. G. Weiss, Butzbach, (*Chemie-Ingenieur-Technik*, 1958, 30, 433). Work was begun at Pintsch Bamag AG on the development of a heavy water production process based on the principle of the chemical two-temperature exchange between hydrogen sulphide gas and water. A part of this work and the planning of a large-scale experimental plant for the production of 0.5 tons  $D_2O$  a year is reported.

### Titanium Production Methods Described by US Mines Bureau

Bureau of Mines experiments in making titanium metal by reducing titanic chloride with sodium are described in a new technical report just released by the US Department of the Interior. The report by V. E. Homme, metallurgist, M. M. Wong, chemical engineer, and D. H. Baker, Jr., supervisory metallurgist, discusses metallurgical studies at the Bureau's Boulder City, Nev., laboratories. The newer experiments with sodium were conducted as part of a continuing Bureau

programme to improve techniques for extracting titanium from its ores.

Equipment and procedures by Bureau metallurgists in reducing titanic chloride with sodium at both low (97.5°C to 200°C), and high (1,000°C) temperatures are described in the report. Reduction was accomplished in two stages with a special reactor.

A copy of Report of Investigations 5398, 'Sodium reduction of titanic chloride', can be obtained from the Bureau of Mines, Publications-Distribution Section 4800 Forbes Street, Pittsburgh 13, Pennsylvania, US.

### Japanese Firms Plan Polythene Production

Two Japanese chemical firms are reported ready to manufacture polythene. At its Iwakuni factory, the Mitsui Petrochemical Co. has built a plant having an annual production capacity of 12,000 tons each of polythene, toluol and xylol, 6,000 tons of ethylene oxide, and 4,800 tons of ethylene glycol. The Sumitomo Chemical Co's factory at Niihama is said to have an annual production capacity of 11,000 tons of polythene.

### Chemical Prospects of Lacq Natural Gas Deposits

A recently completed French Government report on the development of the natural gas deposits at Lacq, South Western France, indicates that SNPA, the producing company, estimate that by 1961 they will be able to offer from the by-products 1.4 million tons of sulphur, 160,000 tons of butane and propane, 100,000 tons of ethylene and 210,000 tons of petrol a year. More than half of this would be available for export, it is stated.

Because of the excess of sulphur available from the US and Mexico, France is understood to be discussing prospects

with the US of the marketing of the Lacq sulphur.

The three development companies already formed to buy essential by-products from SNPA are: Aquitain-chimie, founded by leading French chemical companies including Pechiney and St. Goham and by the Banque de Paris et de Pays-Bas, to produce acetylene and methanol; Azolacq (ONIA Pierrefitte and the Banque de Paris et des Pays-Bas) for fertilisers; and Methanolacq (Aquitainchimie and Kuhlmann) for methanol and all types of chemical product.

### S. African Opposition to US Uranium Extraction Patent

Opposition to the granting of a patent relating to the extraction of uranium applied for by a US company—Kerr-McGee Oil Industries Inc.—is being considered by the South African Chamber of Mines. The invention in question concerns a process for rejuvenation of ion-exchange resins used in extraction of uranium. The process in the US company's invention relates to a method of removal of poisoning ions from the resins.

The UK Atomic Energy Authority has recently developed a technique for extracting uranium from ore which does not employ ion exchange resins.

### Australian Import Duty On Synthetic Resins

Certain decisions on the application of the Australian tariff have been announced by the Australian Department of Customs and Excise. Rate of duty for synthetic resins of the styrene type, being copolymers of styrene and butadiene containing 50 per cent or more of styrene, has been fixed at 7½ per cent *ad valorem*.

## Silica Foams for Insulation

SILICA made to boil and foam is an interesting new insulating and refractory material. Manufactured by Pittsburg Corning Inc., Sedalia, Montana, US, under the name Foamsil, foamed silica is a lightweight, rigid, inorganic block with a closed cell structure. It is produced in a special continuous horizontal furnace. A foaming agent actuates the pure silica which bubbles up into tiny non-interconnecting cells. On cooling these bubbles set.

Of the various properties of the new material, probably the most valuable is its temperature resistance range. Under cyclic temperatures, Foamsil is stated to withstand thermal shock from -450° to 1600°F. At a continuous temperature the limit is 2200°F. Insulating value of 1 inch of the material is claimed to be equal to 18 inches of acid brick at 250°F operating temperature. During rapid temperature changes no warp, shrink or slump has been noted and the material does not crack or spall when subjected to intense heat, such as from an oxy-

acetylene torch and then quenched. At temperatures up to 2200°F, Foamsil expands although its expansion coefficient is low ( $3.5 \times 10^{-7}$  or about one-eighteenth that of low carbon steel).

This new silica foam has a compressive strength of 130 to 210 lb. per sq. in. Pittsburg Corning say that it can be used as a load-bearing surface to eliminate some of the supporting and reinforcing structure usually required with insulation.

Silica foam material can be used for inside linings for tanks and pipelines that contain hot acids, even hot phosphoric acids. Hydrofluoric acid is the exception. Also, the material does not absorb acids, solvents or vapours. Other applications suggested for Foamsil are as lining for concrete, brick or steel stacks subject to corrosive atmospheres and as underground thermal and electrical insulation.

At present Pittsburg Corning supply Foamsil in blocks 11 by 17 in. or 17 by 22 in. in thicknesses up to 3 in. Various insulation shapes can also be obtained.

● The Council of the Royal Society has awarded Mr. and Mrs. John Jaffé Donation Studentships in original scientific research from 1 October, 1958, to Dr. E. A. MAGNUSSON to continue his study of the modification of atomic orbitals in bond formation in the Department of Chemistry, University College, London, and Dr. JENNIFER WILDY to work on the biosynthesis of the protein and haem components of haemoglobin in the Department of Chemical Pathology, University College Hospital Medical School.

● Mr. W. E. BALLARD, managing director of Metallisation Ltd., Dudley, and the author of a book and many papers on corrosion and metal spraying, has been elected president of the Institution of Metallurgists.

● Mr. P. W. FAULKNER, O.B.E., general manager of the chemical and metallurgical division of The Plessey Co. Ltd. since 1953, has been appointed director and general manager of Plessey International Ltd. Mr. Faulkner, who joined Plessey's head office, Ilford, in 1952, remains an executive director, and a director of Technical Ceramics Ltd., a leading company in the piezoelectrics field.

● Mr. N. A. ESSERMAN, B.Sc., F.Inst.P., A.M.I.E. (Aust.), has been appointed the first director of the CSIRO National Standards Laboratory, Sydney. He will continue as chief of the CSIRO Division of Metrology.

● Mr. T. TERTIUS AIKMAN is retiring as chairman of Aikman (London) Ltd. but will remain as consultant to the company.



Mr. Tertius Aikman

The new chairman is Mr. BARRY T. AIKMAN, Mr. Aikman's son. Mrs. Winifred Aikman, Mr. Aikman's wife, is also retiring from the board.

Mr. Tertius Aikman's grandfather started the company in Glasgow in 1830, and the new chairman becomes the fourth generation of Aikmans to head the company. Aikman (London) Ltd., is noted for its half-yearly reports on the nitrogen industry.

● Mr. DENNIS BALMFORTH, B.Sc., A.R.I.C., of Courtauds Ltd., Coventry, has been awarded the Perkins Centenary Fellowship, which is tenable in the Department of Colour Chemistry, Leeds University. He will carry out research on the application of dyestuffs. Perkin Centenary Scholarships have been awarded to Mr. BRIAN HIGGINBOTTOM, assistant chemist, Bleachers' Association Ltd. (tenable at Loughborough College of Technology) and Mr. W. K. McCOURT, laboratory foreman with Courtaulds Ltd., Carrickfergus, N. Ireland (tenable at Queen's University, Belfast). Under a Perkin Exchange Lectureship,

## PEOPLE in the news

short visits will be exchanged between PROFESSOR A. J. BIRCH, F.R.S., Manchester University, and PROFESSOR V. PRELOG, Zurich.

● Mr. C. H. J. CORBETT and Mr. T. WILLIAMS, directors of the Eaglescliffe Chemical Co. Ltd., Stockton-on-Tees, have been appointed joint managing directors. Mr. Corbett's responsibilities will be primarily technical and Mr. Williams' primarily commercial. Mr. Williams will continue his directorship of British Chrome and Chemicals Ltd.

● The name of Mr. V. F. NOEL-PATON, chairman since 1951 of T. & H. Smith Ltd., manufacturing chemists, of Edinburgh, appeared in the first list of life peers to be created. Mr. Noel-Paton was for three years also chairman of Duncan, Flockhart & Co. Ltd.

● Mr. GEORGE WEST has been appointed export manager of the Chemicals Division of Newton Chambers and Co. Ltd., Thorncliffe, near Sheffield, in succession to Mr. C. R. LUND, who died in June. Mr. West, the son of Sir Harold West, former managing director, joined Newton Chambers in 1954. He had experience in the home sales and accounts departments before becoming assistant manager of the export department in March, 1957.

● Mr. A. H. DINGMAN, formerly director of sales, Brunner Mond Canada Ltd., has been appointed director of sales for Brunner Mond Producers by Allied Chemical Canada Ltd. Other appointments made by Allied Chemical, Canada, are: Mr. H. PEERS, formerly sales manager, National Aniline and Chemical Co. Ltd., as director of sales, National Aniline Products; Mr. C. R. BURROWS, formerly Toronto sales manager, Nichols Chemical Co. Ltd., as director of sales, Nichols Products, and Mr. W. H. ROBINSON, formerly assistant to the vice-president, the Barrett Co. Ltd., as director of sales, Barrett Products.

● Mr. J. H. COLLINS, B.Sc., F.R.I.C., consulting chemist and plastics technologist, has come to an arrangement with Priestley Studios Ltd., Gloucester, to move his business address to their Kingsholm Works. He now has facilities to

carry out consulting and development work on plastics materials from earliest design stages to prototype production, especially in the fields of thermoplastics, expanded insulating materials and reinforced plastics. Correspondence can be addressed to him c/o Priestley Studios Ltd., Kingsholm Works, Gloucester (Tel. Gloucester 20206.)

● Mr. ROGER TAYLOR has joined the group advertising department of BTR Industries Ltd., with responsibility for press relations activities.

● Mr. COLIN W. ROBERTSON has been appointed manager of Nederlandsche Dow Maatschappij NV in Rotterdam, a manufacturing and distributing subsidiary of The Dow Chemical Co. in Holland. Mr. Robertson succeeded Mr. JOHN VAN STIRUM, who moves to an executive position with the parent company in Midland, Michigan, US.

● Mr. T. S. LING and Mr. A. C. BUREAU, members of the staff of the Balfour Group, have been appointed directors of Ernest Scott and Co. Ltd., and George Scott and Son (London) Ltd. Mr. Ling is in charge of the Rietz division of



Mr. T. S. Ling



Mr. A. C. Bureau

George Scott and Son (London) Ltd., and Mr. Bureau is director and general manager of Balfour Lecoq Ltd., and gas consultant to Henry Balfour and Co. Ltd.

● THE EARL OF BALFOUR is to become a part-time member of the Scottish Gas Board from 1 September of this year.

● Mr. R. T. HAYES has been appointed a director of United Coke and Chemicals Co. Ltd., a subsidiary of United Steel. Mr. Hayes, who joined the United Steel group in 1946, is commercial manager of United Coke and Chemicals, of United Steel's ore mining branch, and of the Santon Mining Co. Ltd.

● Mr. HERBERT P. BRIDGE has retired, on medical advice, from executive duties in the National Plastics group, but remains a director of National Plastics.

### Obituary

DR. DAVID LEVER, who died on 24 July at his home in Glasgow, was for many years superintendent of the School of Sugar Manufacture at the Royal College of Science and Technology, Glasgow. A former student of the college, he gained his practical experience as chemist to the British Guiana Demerara Co. Ltd.

## Preparation of Pure Sulphur for Physical Measurements

IN connection with a study of the reproducibility of the sulphur boiling point as a fixed point for the International Temperature Scale, R. J. Berry, Division of Applied Physics, National Research Council, Ottawa, Ontario, reports that he has been obliged to consider in some detail the effects of impurities, particularly dissolved gases, on the properties of sulphur. Reference is made (*J. Sci. Instr.* 1958, 35, No. 6, 223) to methods of removing small amounts of residual organic impurity, e.g. by Bacon and Fanelli, who tried alternate freezing, pumping, and melting using a pressure of 1 mm. Hg and found 12 such cycles were necessary to remove dissolved gases; and the method by Malus who reported fresh gas evolved after 80 successive fusions under a pressure of 2 to 3 cm. Hg.

Betty *et al.* have tried the Malus method but with a pressure of  $10^{-3}$  mm. Hg and have found that there is no visible trace of dissolved gas after a single cycle. However, if after degassing the sulphur is allowed to solidify in contact with air an appreciable amount

of gas was evolved when the sulphur was melted.

The solubility of air, nitrogen and helium in sulphur has been examined by these workers. It is reported that from the similarities of the solubilities of nitrogen and air, it was apparent that there was no appreciable chemical reaction between the sulphur and oxygen of the air in the time of the experiment. It was noted that the ratio of the solubilities were similar to the ratio of the solubilities of the same gases in freezing water. The solubilities in sulphur were, however, about seven times less.

As it had been suggested that sulphuric acid was formed in sulphur when stored for long periods, sulphur stored for six months was examined and was found to contain less than 0.001 per cent sulphuric acid.

Results of these experiments indicate that atmospheric gases are dissolved by sulphur to an appreciable extent and can only be removed by fusions under a pressure less than  $10^{-3}$  mm. Hg. Liquid sulphur will remain gas free only while it is under vacuum.

## Cutting Efficiencies of Files Improved by Molybdenum Treatment

TESTS carried out jointly by two companies in the BSA Group, William Jessop and Sons Ltd., and J. J. Saville and Co. Ltd., have shown that molybdenum disulphide treatment of files can improve their cutting efficiency by as much as 60 per cent. A 'dag' dispersion of colloidal molybdenum disulphide was used throughout the tests.

One batch of 12-in. hand bastard files were spray coated with 'dag' Product 1127 (colloidal molybdenum disulphide in alcohol) and others with 'dag' Product 1209 (colloidal molybdenum disulphide in toluene), each diluted with toluene to a suitable spraying consistency. No tempering treatment can be carried out. As it is not possible to heat-cure the coating formed from Product 1209, Product 1127 which requires no subsequent heat-curing

treatment, was used in later tests.

Even better performances were obtained in further tests carried out with 12-in. hand smooth files and 6-in. three square engineers' files, each type having a finer cut than the 12-in. bastard (50 per cent increased efficiency with the smooth and 60 per cent with the three square files). Results revealed that the percentage improvement varied with the fineness of the teeth and was attributed to less clogging with treated teeth.

A valuable 'sideline' advantage claimed for the treatment is the good appearance of the sprayed file, and from preliminary tests carried out the protection the coating provides against corrosion does not appear to be inferior to that offered by materials in current use.

## Newton Chambers Modernise Ensecote Department

THE ENSECOTE department of Newton Chambers and Co. Ltd., at Thorncliffe, near Sheffield, has been modernised and extended to handle materials or structures up to 65 tons in weight and to 120 ft. long. It is now claimed that practically anything that can be transported by road or rail can now be lined with sterile, inert, high-gloss, corrosion-resisting seamless plastics material. In the new pressurised spraying chamber the air, which can be heated in cold weather, is filtered to prevent dust affecting coated surfaces and the newest type of water-washed wall

has been installed to clean and filter the air expelled into the outside atmosphere. Other new plant includes shot-blasting cabinets in which vessels up to 10 ft. in diameter and 40 ft. long can be accommodated.

In addition to introducing new grades of Ensecote and Lithcote for use in the chemical industry and for lining road and rail tanks for transporting chemicals, the department now fabricates equipment from sheet polythene and p.v.c., which are becoming recognised as constructional materials in their own right.

## Chemical Prices Steady in June

WHOLESALE price index of the Board of Trade shows that the figure (provisional) for chemical and allied products in June was 110, the same as in May, 0.3 lower than in January and 1.2 higher than in June last year. The following figures are based on 1954 = 100.

	June, 1957	May, 1958	June, 1958
Chemical & allied products ...	108.8	110.0	110.0
Dyes & dyestuffs ...	111.0	111.1	110.4
Disinfectants ...	112.1	112.9	112.3
Fertilisers ...	119.1	120.4	120.4
Insecticides, weedkillers & fungicides ...	99.9	92.9	92.9
Synthetic resins & plastics materials ...	94.5	94.7	94.6
General chemicals ...	106.5	107.9	107.3
Benzole, pure, BSS 136-1950 ...	102.1	106.9	106.9
Caustic soda liquor, 100° TW ...	108.7	116.3	116.3
Soda ash, light (delivered) ...	108.8	115.3	115.3
Soda ash, light, f.o.r. works ...	109.9	117.7	117.7
Sulphuric acid, BOV ...	114.4	103.9	103.9
Sulphuric acid, ROV 94/95 per cent ...	106.0	104.7	104.7
Drugs & pharmaceuticals ...	98.7	98.1	98.0
Synthetic detergents ...	101.3	103.1	103.1
Ethyl alcohol, industrial, BSS 507-1933 ...	164.4	146.2	146.2
Commodities wholly or partly imported			
Pyrites, c.i.f. UK ports ...	102.4	76.5	72.7
Sulphur, crude (for acid making) c.i.f. ...	116.1	84.2	83.5

## British Gas Statistics

Industrial sales of gas for the year ended March, 1958, were about 5 per cent higher than in 1956/57, while commercial sales were practically unchanged. Five per cent less coal gas was made and the amount of coal carbonised was over 1½ million tons less. The water gas made was higher by 39 per cent, and oil gas increased by 11 million therms to 16 millions therms in 1957/58. Surplus gas purchased from oil refineries and coke ovens increased by 6 per cent. Crude tar production fell by 3.4 per cent to 1,921,000 tons and benzole production by 5.1 per cent to 27,800,000 gall.

## More Pure Science and Technology Students in 1956-7

The number of full-time students in British universities and university colleges increased from 85,194 to 89,866 in the year 1956-7. A greater expansion is expected to be shown in statistics for 1957-8. Of these students 43.1 (43) per cent studied arts subjects and 22.2 (21.3) per cent pure science. The number of medical students dropped by 1.3 per cent to 14.4 per cent, but technology students increased from 13.3 to 13.9 per cent. In the same year recurrent expenditure increased to £41.5 million (£38 million).

## Cheaper Stainless Steel

The price of ferritic stainless steel strip known as FI.17 in sizes up to, but not including, 15 in. wide is to be reduced by £15 to £35 per ton according to thickness—the first reduction in the price of stainless steel since the war, says Firth-Vickers Stainless Steels Ltd., Sheffield. Type FI.17 is mainly used in the motor car industry.

## Commercial News

### British Tar Products

Total profit of British Tar Products for the year to 31 March 1958, was £206,696, compared with £110,579 for 1956-7. Commitments totalled £70,000. An outlet has been found for surplus funds in a very substantial expansion of storage installation.

Fixed assets have been shown by an independent valuation in 1957 on a replacement basis to be valued at about £750,000, excluding land, tank wagons, house property, etc., against relative book value of £182,059.

Mr. F. Woolley-Hart, chairman, referring to trade investment income from Benzole Producers, comprising £45,732 from assets sales and £31,385 dividend covering a period of years, pointed out that the operations of this company were now considerably limited; it was not possible to forecast future receipts from that source.

### Hickson and Welch

A scrip issue of three 10s ordinary shares for every seven held is announced by Hickson and Welch (Holdings). The new shares will rank for the final dividend in respect of the current year.

Group profits to date are stated to be similar to those for the corresponding period of last year.

### Fisons Pty. SA

Partial finance of Fisons' new factory at Sasolburg, SA, has been completed by arrangement with the Industrial Development Corporation, the Commonwealth Development Finance Co., and the Industrial Finance Corporation of South Africa.

The new factory, scheduled to produce sulphuric acid, superphosphate and compound fertilisers as from next year, involves loan and preference capital of £1,850,000.

### Edwards High Vacuum Ltd.

According to a prospectus issued by Edwards High Vacuum Ltd., ordinary shareholders on the register at 21 July are offered £450,000 6½ per cent convertible unsecured loan stock 1978-83 at £99, on the basis of one stock for five ordinary shares. The offer closes on 15 August.

### ICI of Australia and NZ

For the year ended 30 September, 1957, the profit of Imperial Chemical Industries of Australia and New Zealand Ltd. dropped by approximately £110,000 to £2,544,501. Net profit was some £40,000 lower at £2,190,857, but dividends increased from £1,143,500 to £1,216,400. Total sales by the company and its subsidiaries were up by £1,248,000 to £37,971,000. Sales of the alkali products group were higher than

in the previous year and the alkali works at Osborne, SA, are being extended. The 'Biazzì' type continuous process nitroglycerine plant at Deer Park had produced in its first year of operation a high quality product.

### Benn Brothers Ltd.

The directors of Benn Brothers Ltd. recommend the payment of the following final dividends, less tax, for year ended 30 June: 3 per cent on preference, which with interim of 3 per cent paid in February, makes 6 per cent; and 10 per cent on ordinary, which with interim of 5 per cent paid in February, makes 15 per cent (same).

### Ugine

Manufacturers of electro-chemical, electro-metallurgical and electric steel products, Ugine, Paris, France, maintained progress. Turnover increased from Frs.45,502 million in 1956 to Frs.55,151 million. After appropriations of Frs.3,468,159,309 for depreciation and new investments, net profits amounted to Frs.1,557,095,635. It was therefore proposed to distribute a net dividend of Frs.410 per share.

The report indicates that there was a strong demand for magnesium, output rising to 1,500 tons. Production increased in the field of fluorine products and their chlorine derivatives as well as ferro-alloys, abrasives, powder metallurgy and permanent magnets. Aluminium production also increased following the first full year of operation of the Venthon plant. Sales amounted to 30,000 metric tons.

Forming part of Ugine's current modernisation and expansion programme is the manufacture of aluminium in south western France using power generated with natural gas from the Lacq deposit. Other projects in hand are intended to ensure Ugine's supplies of new materials and include the surveying of nickel and chrome ore

deposits in Madagascar with a view to commencing production in the near future.

### Griffiths Hughes

Indications that costs have ceased to rise, at least in some directions, should mean an end to the squeezing of profit margins, says Mr. C. B. Green, chairman of the Griffiths Hughes Proprietaries Group, in his annual report.

Group profits of £439,921 for the year ended 31 March, 1958, compared with £465,659. The substantial increase in sales and the effecting of further economies were not sufficient to offset the rise in costs.

The year's dividend, however, is to be increased from 7½ to 10 per cent, and the directors would not have favoured this if there had appeared any prospect that it might have to be cut back again in the foreseeable future. The acquisition of the J. C. and J. Field business was not likely to make a material contribution to the group profit in its first year, since their activities were being integrated with those of the parent concern.

The consolidated balance sheet, excluding the new Field subsidiary, shows fixed assets of £788,895, as against £784,966, and current assets of £1,477,592, as against £1,563,072, and liabilities of £608,773 as against £547,359.

### INCREASES OF CAPITAL

D. McDERMOTT (CHEMICALS) LTD., 31 Gladstone Street, Widnes. Increased by £6,000 beyond the registered capital of £14,000.

WRIGHT, LAYMAN & UMNEY LTD. Manufacturing chemists, 46 Southwark Street, London SE1. Increased by £10,000 beyond the registered capital of £370,000.

PHARMETHICALS (LONDON) LTD., industrial and manufacturing chemists, etc., 20 Gerrard Street, London W1. Increased by £32,500 beyond the registered capital of £10,000.

### Market Reports

### PRICES GENERALLY WELL HELD

**LONDON** Quiet trading conditions have been ruling on the general chemicals market, and the usual seasonal contraction in deliveries to the consuming industries has been in evidence. Nevertheless the demand is reasonably good for the period and prices continue unchanged and firm. The flow of export inquiry has been well maintained. Allowing for seasonal influences the demand for coal tar products, on home account, has been steady.

**MANCHESTER** Prices generally on the Manchester market for heavy chemical products have been well held during the past week. There has been no indication of the reaction which a number of consumers, judging by their cautious buying policy, seemed to expect. Apart mainly from textile chemicals, which are meeting with a more restricted demand, there

is a fair contract movement of the alkalis and other products on the home market, with shipping business running at about the level of recent weeks. In the fertiliser section, there has again been some forward buying in those lines where early delivery rebates operate, but otherwise the demand remains seasonally quiet.

**GLASGOW** As reported last week the Glasgow Fair holidays have had their effect on the Scottish heavy chemical market, where trading generally during the past week was very quiet. From areas not affected by holidays, conditions were fairly active. Little or no change in prices has taken place. Although not quite so active, there is still a reasonable demand for agricultural chemicals.

# NEW PATENTS

By permission of the Controiler, HM Stationery Office, the following extracts are reproduced from the 'Official Journal (Patents)', which is available from the Patent Office (Sale Branch), 25 Southampton Buildings, Chancery Lane, London WC2, price 3s 3d including postage; annual subscription £8 2s.

Specifications filed in connection with the acceptances in the following list will be open to public inspection on the dates shown. Opposition to the grant of a patent on any of the applications listed may be lodged by filing patents form 12 at any time within the prescribed period.

## ACCEPTANCES

Open to public inspection 3 September

Process for enrichment of water in deuterium oxide. Urey, H. C., and Grosse, A. V.

- Separation of uranium from thorium. Fontana, B. J. **800 730**
- Method of producing uranium pentachloride. McCombie, H. R. **800 791**
- Preparation of diborane. Schelsinger, H. I., and Brown, H. C. **800 731**
- Dual temperature isotope exchange process. Joris, G. G. **800 732**
- Extraction of liquid, such as water, from air or other gases. Godfrey & Partners (Holdings), Ltd., Sir G. **800 851**
- Apparatus for the treatment of liquids. Sutcliffe, Speakman & Co., Ltd., and Edwards, F. E., [Personal representative of Edwards, W. E. (deceased)]. **800 932**
- Extracting precious metals from silicates. Compagnie Francaise de Metallurgie Aurifere. **801 011**
- Treating residual liquors obtained by the distillation of alcohol from mash. Young, R. A. G. **800 809**
- Production of high molecular weight polyethylenes. Ziegler, K. **800 935**
- Vat dyestuffs of the benzanthrone-pyrazole-anthrone series. Farwerke Hoechst AG. **801 031**
- Manufacture of amino-triaryl-carbinols and triaryl-methane dyestuffs. Ciba Ltd. **800 971**
- Method and apparatus for the discharge of molten material from gas producers or the like. Kohlenscheidungs-G.m.b.H. **800 857**
- Esters of phenolformaldehyde resins and process for preparing same. General Mills, Inc. **801 812**
- Sulphur-containing benzoxazole derivatives and compositions containing them. Boots Pure Drug Co., Ltd. **800 998**
- Electrical insulating materials composed of high molecular weight polycarbonates. Farbenfabriken Bayer AG. **800 713**
- Methods and apparatus for producing uranium fluoride. Commissariat A l'Energie Atomique. **800 815**
- Pills or tablets. Sterling Drug, Inc. **800 692**
- Liquid-Flow control systems. Plessey Co., Ltd. **800 973**
- Filter arrangements. Neidig, A., Neidig, W., Neidig, W., Neidig, H., and Neidig, W., [trading as Neidig Söhne Maschinenfabrik, F. A.]. **801 051**
- Vaporisation of water and light oils from tar. Drake-Didier-Kogag, Ltd. (Didier-Kogag-Hinselmann G.m.b.H.). **801 003**
- Treatment of surfaces. Imperial Chemical Industries, Ltd. **801 014**
- Pour point depressant for lubricating oils. Continental Oil Co. **800 714**
- Hydrocarbon sweetening process. Standard Oil Co. **800 693**
- Silicas. Columbia-Southern Chemical Corp. **800 866**
- Acylamino-amino-anthraquinones and a process for their production. Farbenfabriken Bayer A.G. **800 694**
- Diquaternary compounds and the manufacture thereof. Wellcome Foundation, Ltd. [Cognate applications 22 376 and 22 377]. **800 962**
- Method of preparation of metal salts. Hall, J. D. **800 695**
- Ammonium nitrate and compositions containing it. Imperial Chemical Industries. **801 068**
- Ductile chromium and chromium rich alloys. General Electric Co. **800 794**
- Production of diketene. Distillers Co., Ltd. **801 032**

- Substituted amino-guanidines. Imperial Chemical Industries, Ltd. **800 869**
- Combustion chamber liquid-fuel flow control means. Bendix Aviation Corp. **800 872**
- Polymerisation process. Distillers Co., Ltd. **800 873**
- Separation of pyrochlore from calcite, apatite and silica. Burks, H. G. **800 717**
- Substituted alkylamines and processes for their production. Philips' Gloeilampenfabrieken, N.V. **800 718**
- Production of N-acyl taurides. General Aniline & Film Corp. **800 827**
- Production of esters. Distillers Co., Ltd. [Cognate application 239.] **800 875**
- Sulphonium compounds. Beecham Research Labs., Ltd. **800 963**
- Lubricating oil additive compositions and lubricating oil compositions containing them. Esso Research & Engineering Co. **800 964**
- Production of modified carbon black. Deutsche Gold- und Silber-Scheideanstalt Vorm. Roessler. **800 975**
- Treatment of light metals. Kaiser Aluminium & Chemical Corp. **800 879**
- Production and use of oxidation catalysts. Coal Tar Research Association. **800 783**
- Textile colouring process. Imperial Chemical Industries, Ltd. **800 735**
- Steroid compounds. Pfizer & Co., Inc., C. **800 797**
- Horticultural pest control agents. Lumb, M., and Duddington, C. L. [Cognate applications 16878 and 33102.] **800 736**
- Production of thermoplastic compositions. Imperial Chemical Industries, Ltd. **800 744**
- Water-insoluble salts of streptomycin antibiotics and pharmaceutical preparations containing same. Bristol Laboratories, Inc. **800 738**
- Sweetening sour hydrocarbon distillates. Universal Oil Products Co. **800 721**
- Production of melamine from urea. Montecatini Soc. Generale per l'Industria Mineraria e Chimica. **800 722**
- Moisture separator. Specialties Development Corp. **801 054**
- Degradation of polyethylene terephthalate to dimethyl terephthalate. Vereinigte Glanzstoff-Fabriken AG. **800 697**
- Wet screening of solid particles and apparatus therefor. Stamicarbon N.V. [Addition to 791 520.] **800 745**
- Detergent compositions. Hedley & Co., Ltd., T. **801 018**
- Methods for producing concentrated superphosphates. Lumms Co. **800 698**
- Esterification of cellulose and apparatus therefor. Gevaert Photo-Producten N.V. **800 746**
- Siliceous pigments. Columbia-Southern Chemical Corp. **800 748**
- Mono- and di-alkyl anhydrotetracyclines. Pfizer & Co., Inc., C. **800 699**
- Method of obtaining sodium salts of barbituric and thiobarbituric acid derivatives. Soc. Financiere de Placements, Soc. Anon. **800 886**
- Emulsions for making moisture impermeable paper and the like. Patent & Licensing Corp. **800 750**
- Heterocyclic ketones. Dann, O. **800 751**
- Meta-sulphides of polyhalogenated phenols. Farbenfabriken Bayer AG. **800 752**
- S-acyl-pantetheins and process for the production thereof. Bracco, F. **800 701**
- Organosilicon compounds. Midland Silicones, Ltd. **800 754**
- Separating and recovery of purified di-tertiary-butyl-para-cresol. Koppers Co., Inc. **800 702**
- Production of cyanuric chloride. Lonza Electric & Chemical Works, Ltd. **800 703**
- Process for the simultaneous production of acetylene and a mixture of nitrogen and hydrogen. Texaco Development Corp. **800 888**
- Preparation of 3:4-dichlorobutene-1. Distillers Co., Ltd. [Addition to 798 393.] **800 787**
- Steroids. Upjohn Co. **800 889**
- Apparatus for melting metals, more especially titanium, in an arc with a self-consuming electrode. Deutsche Gold- und Silber-Scheideanstalt Vorm. Roessler. **800 966**
- Hydrogenation of aliphatic mono- and dicarboxylic acids. Vereinigte Glanzstoff-Fabriken AG. **800 847**
- Acid milling of vatable organic compounds. Fairweather, H. G. C. **800 784**
- Polyurethane elastomers. Du Pont de Nemours & Co., E. I. **801 023**
- Production of cyclohexane. Esso Research & Engineering Co. **800 983**

- Liquid detergent compositions. Colgate-Palmolive Co. **800 706, 800 785**
- Sweetening petroleum naphthas. Standard Oil Co. **800 984**
- Preparation of dichloro difluoromethane. National Smelting Co., Ltd. **800 759**
- Liquid adhesive or other viscous liquid dispensers. Dachinger, H. **800 707**
- Production of high boiling propylene polymers employing liquid phosphoric acid catalyst. California Research Corp. **800 708**
- Liquid filters. General Motors Corp. **801 054**
- Continuous production of polyamides from dicarboxylic acids and diamines. Vereingigte Glanzstoff-Fabriken AG. **801 058**
- Moulding compositions of high molecular weight and low molecular weight polymers. Dow Chemical Co. **801 036**
- Steroids and the manufacture thereof. Upjohn Co. **801 024**
- Pyrimidine sulphonamide and a process for the manufacture thereof. Hoffmann-La Roche & Co., AG. **800 776**
- Preparation of orotic acid. Soc. Des Usines Chimiques Rhone-Poulenc. **800 709**
- Quaternary ammonium compounds. Rohm & Haas G.m.b.H. **800 830**
- Method for the manufacture of tubes and rods of desired cross-section made of fused silica. Quartz & Silice. [Addition to 772 826.] **801 025**
- Process and plant for separating particles of solid material into fractions according to specific gravity. Stamicarbon N.V. **800 906**
- Purification of acetone. Distillers Co., Ltd. **800 763**
- Detergent compositions. Unilever, Ltd. **800 710**
- Therapeutic product. Bristol Laboratories, Inc. **801 026**
- Preparation of sulphonyl chlorides. Fusco, R., and Bianchetti, G. **801 037**
- 2-Hydroxy-benzo [a] quinolizines, acid addition salts thereof and a process for the manufacture of same. Hoffmann-La Roche & Co., AG., F. **800 969**
- Fertilisers containing ammonium nitrate, dicalcium phosphate and calcium sulphite. Stamicarbon N.V. **801 027**
- Indole derivatives and process for their preparation. Soc. Des Usines Chimiques Rhone-Poulenc. **800 765**
- Apparatus for use in wet screening solid particles. Stamicarbon N.V. [Addition to 791 520.] **801 028**
- Method for aromatisation of light naphthas. Esso Research & Engineering Co. **801 060**
- Phenol-formaldehyde resin and process of making same. General Mills, Inc. [Divided out of 800 998.] **800 999**
- Unsaturated acyl isothiocyanates and the production thereof. Farbenfabriken Bayer AG. **800 887**
- Pyrimidine derivative and a process for the manufacture thereof. Hoffmann-La Roche & Co., AG. **800 777**

## ACCEPTANCES

Open to public inspection 10 September

- Recovery of plutonium. Seaborg, G. T., and Willard, J. E. **801 379**
- Preparation of tetravalent uranium compounds. Benning, A. F., and Benner, R. G. **801 380**
- Production of uranium deuteride. Newton, A. S. **801 094**
- Production of uranium dioxide masses. UK Atomic Energy Authority. **801 381**
- Production of uranium tetrafluoride. UK Atomic Energy Authority. **801 382**
- Fatty products. Unilever, Ltd. **801 118**
- Hydrocatalytic desulphurisation of petroleum hydrocarbons. British Petroleum Co., Ltd., Porter, F. W. B., and Northcott, R. P. **801 385**
- Production of polyethers. Distillers Co., Ltd. **801 429**
- Plasticised compositions. Distillers Co., Ltd. **801 430**
- Production of niobium pentachloride. UK Atomic Energy Authority. **801 386**
- Production of alcohol by fermentation. Genatosan, Ltd. **801 274**
- Photo-sensitising dyestuffs and their manufacture and products containing them. Farwerke Hoechst AG. **801 275**

## PUBLICATIONS RECEIVED

- Precision Tube Developments: Accles & Pollock, Ltd., Oldbury, Birmingham.
- Automatic Control Valves for all Purposes, MCV/43: The Magnetic Valve Co., Ltd., 28 St. James's Place, London, S.W.1.
- Paramins: Esso Petroleum Co., Ltd., 36 Queen Anne's Gate, London, S.W.1.

## Silicates and the Egg



CONSIDER the shell of an egg. As strong as it need be, it is a perfectly adapted container in itself. Although not used in the production of this particular container, silicates do play a major part in the manufacture of the corrugated board cases which are being increasingly used by egg packers. For silicate bonded board has that added strength and rigidity needed for packaging and transporting eggs in bulk.

Once again, silicates lend a hand. But whatever the job, you can be sure that Crosfield, with their experience of the many and varied uses of silicates, will be ready to help. Why not take your enquiries to Crosfield?

**CROSFIELD**  
**SUPPLY SILICATES**  
**FOR**  
**INDUSTRIAL USE**

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JOSEPH CROSFIELD & SONS LIMITED, WARRINGTON, LANCASHIRE

CSP 40-8058-120

## TRADE NOTES

### Nickel in Oxide Sinter

The Mond Nickel Co. Ltd. announce a reduction of £20 in their price for oxide sinter per ton of nickel content, bringing the price to £550 delivered UK works. The reduction is in line with the announcement made on 23 July by The International Nickel Co. of Canada Ltd.

### Du Pont Odourless Bleach

A compound has been developed for bleaching fabrics during dry cleaning which is said to be quite odourless. It may be used, the makers claim, for the treatment of even the most delicate fabrics. The material made at present in only experimental quantities, is designed for use in either dry bleaches or cleansers. The compound, called 'Oxone' monopersulphate, is manufactured by E.I. du Pont de Nemours and Co., whose British representatives are at Bush House, Aldwych, London WC2.

### B & K Labs. New Service

New premises acquired by B & K Laboratories at Tilney Street, Park Lane, London W1, will be formally opened during September to provide a permanent show of international developments in electronic instrumentation, a cinema and an extensive library compiled from worldwide sources.

The work of order processing, servicing and shipping will continue from Union Street, London SE1, but as from September sales enquiries will be dealt with from 4 Tilney Street, Park Lane, London W1, Grosvenor 4567.

### Timber Protection

To develop the use of boron compounds for the protection of timber and plywood against insect pests and fungi, Borax Consolidated Ltd., announce the opening of a timber department under Mr. D. R. Carr, B.Sc., recently wood preservation officer for the New Zealand Forestry Service.

### Chilean Nitrates Prices

New prices of Chilean nitrate of soda and Chilean potash nitrate for the season 1958-9 come into operation on 1 August, state the Nitrate Corporation of Chile Ltd.

Agricultural prices for lots of not less than 6 tons delivered carriage paid to any station in Great Britain or c.i.f. Isle of Man ports are as follows:

	Nitrate of Soda		Potash Nitrate	
	16% N.		15% N. and 10% K <sub>2</sub> O	
August/September	£26	10 0	£29	10 0
October/November	£26	15 0	£29	15 0
December/February	£27	0 0	£30	0 0
March/June	£27	5 0	£30	5 0

The industrial price for refined granulated Chilean nitrate of soda, over 98 per

cent, in lots of 6 tons or more delivered carriage paid to any railway station in Britain is £29 net per ton. There is an allowance of 15s per ton for collection from any nitrate depot.

### New Gritting Emulsion

A new emulsion with unusual properties for roof treatment, to be known as Bituproof 783 Gritting Emulsion, has been introduced by British Bitumen Emulsions Ltd., of Slough, Bucks. Cold applied by brush to the roof surface, the emulsion, on contact with the gritting chips, precipitates the bitumen to give

an immediate protective and adhesive coating which is unaffected by rain. The elimination of pre-heating avoids delays, and the emulsion can be used in adverse weather conditions, so saving time and labour costs.

### Data Sheets

A new series of technical data sheets issued by Johnson Matthey and Co. Ltd. covers their ranges of high temperature platinum-wound electrical resistance furnaces and also JMC furnace control units. The data sheets may be obtained by those not already on the company's mailing list for high temperature furnace information from the head office at 73/83 Hatton Garden, London EC1.

### Stocks and Shares

## Markets Remain Steady Despite International Situation

OVER the last fortnight markets having fluctuated a considerable amount have recently shown much more confidence. A main reason for activity has been that Wall Street and Sterling have shown strength in face of the international political crisis. Consequently gilt-edged and industrial shares have emerged to their pre-Iraq level.

Two factors are now playing an important part in setting the tone of markets. First, with the continued strength in sterling, particularly at this normal period of seasonal weakness, the official rate against the dollar is above par. At this stage it can only be suggested that a gradual lowering of interest rates this autumn is inevitable.

The second factor influencing markets at present is the behaviour of Wall Street. The effect of the Middle East situation on US investors has been to stimulate further their confidence that the recession is over and that inflation will soon succeed it.

British Oxygen having announced an unchanged interim dividend of 4 per cent in respect of the year to 30 September, 1958, poses the question of a suitable investment. The company (registered in 1886) has been mainly concerned with producing oxygen, hydrogen, nitrogen, dissolved acetylene and other gases, and the manufacture of plant and apparatus connected with production and use of its gas products. Numerous factories are scattered at home and abroad. Last year sales expanded in all overseas countries, in some by as much as 25 per cent and enhanced outputs aided a general and considerable improvement in earnings.

Latest reports indicate that the company is continuing to push forward very satisfactorily.

In the table below it is shown that although profit margins have remained steady despite rising sales, earnings on capital employed have failed to keep pace.

### Record of Expansion

	1950	1957
Sales (£m.)	17.8	42.5
Trading profit (£m.)	4.3	10.2
Gross profit margin (% of sales)	24.3	24.1
Return on average capital employed (%)	17.5	14.0
Earnings on Ordinary capital (%)	22.1	24.2
Ordinary dividend	8.9	10.0

There are signs that the ordinary shareholder may in future do rather better from any further expansion in the business. Earlier this year the chairman stated that the directors had considered 'most carefully whether the increase in available profit would enable them to recommend some increase in ordinary dividend.' At least this shows the board has the claims of equity shareholders well in mind, and a higher dividend is clearly on the cards for the future. At about 38s. on their 10 per cent dividend they yield just over five per cent.

1958		Security	July 29	Change on week
High	Low			
17/6	13/4	Albright & W. 5/-	16/10	-3d
20/7	11/9	Bakelite 10/-	18/9	-3d
20/4	14/10	Borax Dfd. 5/-	16/6	-7 1/2d
15/-	10/4	Bt. Glues 4/-	13/10	-4 1/2d
6/-	5/-	B.I.P. 2/-	5/10	—
41/-	28/3	Bt. Xylonite	38/6	+1/6
50/6	45/9	Fisons	49/4	+4 1/2d
40/1	31/6	Glaxo 10/-	40/1	+10 1/2d
41/3	31/-	Hickson & W. 10/-	41/3	+3/9
30/4	24/3	ICI	29/-	+3d
3/4	2/7	Kleemann 1/-	3/1	—
18/-	14/-	Laporte 5/-	16/10	-1 1/2d
16/1	12/6	Monsanto 5/-	14/6	-4 1/2d
13/3	10/10	Reichhold 5/-	13/3	+1 1/4d



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